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Egyptian mummification**

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**Chemical Investigations of the Organic Embalming Agents
Employed in Ancient Egyptian Mummification**

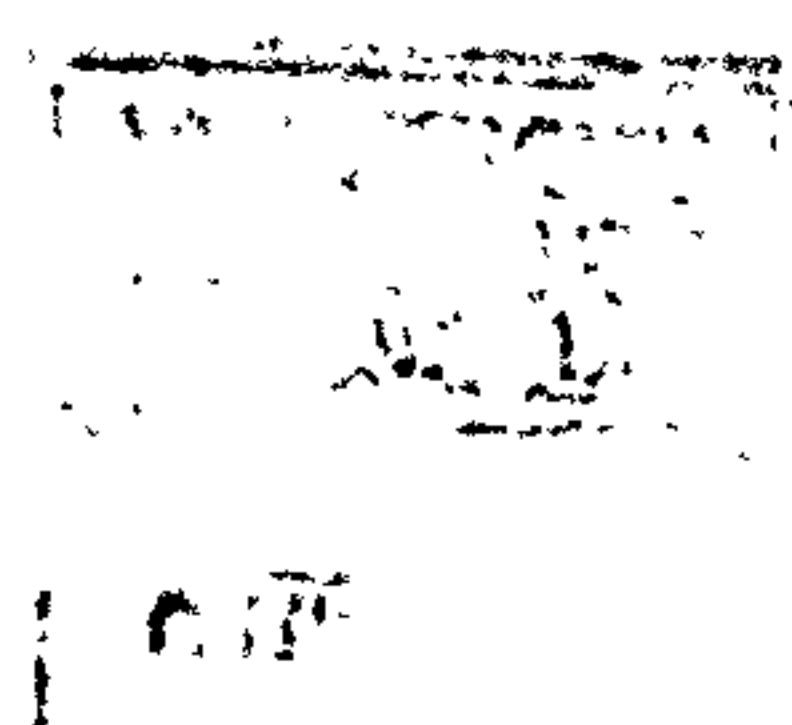
by

Stephen Andrew Buckley

January 2001

This thesis is submitted to the Faculty of Science of the University of Bristol, UK in
fulfilment of the requirements for the degree of Doctor of Philosophy.

Word Count:
79,577



ABSTRACT

This thesis concerns the chemical analysis of organic embalming materials employed in ancient Egyptian mummification. The characterisation and identification of biomarkers present in the samples of packing, wrappings, tissue and 'resinous/bituminous' material analysed has provided valuable archaeological information concerning commodity use, and an assessment of biomolecular transformations occurring over archaeological timescales.

Given the complex nature of the organic materials likely to have been employed in ancient Egyptian mummification, a dual analytical approach was employed. Sequential TD-GC/MS and Py-GC/MS, in addition to the more conventional GC/MS following solvent extraction procedures facilitated the chemical characterisation of both the free and polymeric/bound components of the organic embalming agents employed. The small sample sizes (<0.1mg) required for sequential TD-GC/MS and Py-GC/MS is particularly attractive given the sensitive nature of human remains.

The main components seen in all the mummies were degraded acyl lipids, derived from plant oils and animal fats, many having undergone complete hydrolysis of the ester linkages in the original lipids (e.g. TAGs). The majority of samples analysed were also highly oxidised, with evidence for autoxidation and, more unusually, enzymic oxidation of the fatty acids and terpenoid compounds present. In addition, extensive polymerisation of the fats/oils and resins was evident in many of the 'balms'.

The systematic investigation of the provenanced and dated mummies investigated in this study has allowed meaningful inferences to be drawn on chronological, human/animal and anatomical aspects of Egyptian mummification. Animal mummies were found to have been treated with commodities such as resins (diterpenoid, such as those of the *Pinaceae* family, and triterpenoid, such as *Pistacia* sp.) which would require importation into Egypt. These included cedar resin, identified unequivocally (by the presence of characteristic benzocycloheptenes) in an Egyptian mummy for the first time. Its use, which it has been suggested was not economically viable 'even' for human mummification, clearly dispels the widely held myth that animals were subjected to inferior treatments by the Egyptian embalmers.

The identification and apparent use of Chinese insect wax [characterised by long chain wax esters (C_{48} to C_{60})] in a Ptolemaic mummy would appear to confirm that trade links were established with China at that time, although it must be stated that at this post-Alexandrian date this is not unexpected. Bitumen was not found to be present in any of the mummies (the steranes and hopanes diagnostic of natural bitumens were absent from all the samples analysed), despite the widespread use of the term 'bitumen' to describe organic embalming materials. Interestingly, the use of significant amounts of beeswax, evidenced by the C_{25} to C_{33} *n*-alkanes, C_{40} to C_{50} wax esters, and C_{42} to C_{54} hydroxy wax esters, in most of the mummies analysed is consistent with the ancient Egyptian-derived word for wax 'mum' rather than the usually presumed derivation of 'mummy' from the Persian word for bitumen.

ACKNOWLEDGEMENTS:

I would like to acknowledge the help and support given to me by the following individuals and institutions:

Professor Richard Evershed and the University of Bristol are thanked for their support in providing the laboratory facilities, consumables and travel funds necessary for undertaking my research. A very special thanks, for providing the majority of the funding, must go to Mr Clifford Buckley and, in particular, Mrs Lillian Buckley, without whom this research would not have been possible.

Dr. Kate Robson Brown of the Archaeology Department and staff and students of the School of Chemistry, including Dr. Andrew Stott, Gordon Docherty, Robert Berstan, Christopher Nott, Susan Jim, Dr. Helen Talbot, Dr. Simon Woodbury, Dr. Stephanie Dudd, Dr. Pim van Bergen, Professor James Maxwell, Dr. Graham Nickless, Dr. Dave Roberts, Professor Neil Connolly, Dr. Matthew Flannery, Dr. Kath Ficken, Helen Bland, Luke Avsejs, Mark Copley, Zöe Crossman, Vicky Jones, Paul Chamberlain, Sophie Aillaud, Mark Howland, Alex James, Simon Magness, Dr. Andrew Govenor, Dr. Andrew Turner, Dr. Neil Crawford, Julie Scott, Filip Volders, Dr. Matthew Lockheart, Dr. Hazel Mottram, Dr. Mohammed Elhmmali, Jim Carter, Andy Gledhill, Matt Smith, Tony Court, Sue Trott, Dr. Ian Bull and my advisor, Professor Richard Evershed, for his advice and support (and for recognising when my pedantry is crucial, and when, perhaps, it is not!).

Thanks to everyone in W309 and W403 for both their help and advice, and making my time in Bristol fly by (others may disagree!).

Special thanks to Dr Andy Stott, Rob Berstan, and Chris Nott for testing my liver to its limits. Thanks(?) also to Gordon Docherty for his numerous references to pink (which perhaps point to his own obsession!) and Rob (again) for starting the “pink thing” with his pink (not red!) top. Matt Flannery is thanked for his kind hospitality in providing a (separate!) bed for the night after being ‘the last man standing’ (to use a Matt type phrase) at number of his excellent parties. Thanks to the affable Ian Bull for fully appreciating the nuances of my philosophical outlook on life, and Matt ‘southern ponce and proud of it’ Lockheart for not even trying to. Dr Kath Ficken is thanked for her open-minded, non-dogmatic approach to our many ‘discussions’, and Susie is thanked for showing me that there is another (albeit very long!) way. Thanks also go to Mohammed for his advice on both Bond Elut columns and the dangers of drink!

Sue Giles of the Bristol Museum & Art Galleries; Dr. Margaret Serpico of University College, London; Dr. John Taylor and Dr. Jeffrey Spencer of the British Museum, London; Carla Galorini of the Egypt Exploration Society, London; Dr. Robert Foley and Maggie Bellatti of the Duckworth Collection, Cambridge University; Alison Walster of Weston Park Museum, Sheffield; Professor Don Brothwell of York University; Joanna Hayward, Tracey Seddon and Siobahn Watts of Merseyside County Museums, Liverpool; Dr. Rosalie David of Manchester University Museum; Dr. Elizabeth Goring, Dr. Bill Manley, Dr. Kathy Eremin, Lesley-Ann Liddiard, Dr. Anita Quye, Brian Melville, Dr. Jim Tate of the Royal Museum of Scotland, Edinburgh; Roxie Walker and Dr. Joann Fletcher of the Bioanthropology Foundation; Diane Bergman of the Wilbour Library, Brooklyn Museum; Professor Roger Smith and Loughborough University are thanked for providing me with the initial opportunity to carry out my research ideas prior to my doctoral research at Bristol; Dr. Mohammed Nasr, Dr. Mohammed el-Saghir and Dr. Nur el-Dinn, Supreme Council of Antiquities; Hassan Ibrahim Abu el-Haggag and family of Qurna.

For their support and encouragement, Lesley Allen; David Beaumont; David Buckley; Jacqueline Buckley; Ian Buckley; Alice Dawson; Lillian Buckley and of course Jo.

To Mum and Jo who made it all possible.

“Believe those who are seeking the truth; doubt those who find it”.
- Andre Gide, 1959

“The average PhD thesis is nothing that the transference of bones from one graveyard to another”
- J. Frank Dobie, 1945

“Speak! for thou long enough hast acted Dummy.
Thou hast a tongue – come – let us hear its tune;
Thou’rt standing on thy legs, above-ground, Mummy!
Revisiting the glimpses of the moon,
Not like thin ghosts or disembodied creatures,
But with thy bones and flesh, and limbs and features.”
- H. Smith, 1846

DECLARATION

I hereby certify that the work described herein is my own, except where otherwise stated, and has not previously been submitted for examination at this, or at any other, University. Any views expressed in the thesis are those of the author and in no way represent those of the University of Bristol.

Stephen A. Buckley

Stephen A. Buckley

Addendum

Since the submission of this thesis further work carried out at Bristol has shown that the identification of a sesquiterpenoid as tetramethylhexahydrobenzocycloheptadiene in a mummified cat is incorrect, and so the 'unequivocal' identification of cedar resin can no longer be justified. However, the two oxidised sesquiterpenoids, identified as 'tetramethylhexahydrobenzocycloheptadienone' and 'tetramethyltetrahydrobenzocycloheptatrienone' are consistent with the oxidised nature of the diterpenoid acids, 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid observed in these samples, with only a trace amount of the dehydroabietic acid biomarker surviving. Further research would be needed in order to provide the unequivocal identification of cedar resin in this mummy, namely confirmation of the oxidised sesquiterpenoids via co-injection studies of oxidised modern cedar oil/resin.

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ABBREVIATIONS AND NOMENCLATURE

AAS	atomic absorption spectroscopy
BSTFA	N,O-bis(trimethylsilyl)trifluoroacetamide
DAG	diacylglycerol
DKP	diketopiperazine
FA	fatty acid
FTIR	fourier transform infra-red
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
GCC/IRMS	gas chromatography combustion isotope radio mass spectrometry
IR	infra-red
MAG	monoacylglycerol
MAIG	monoalkylglycerol
<i>m/z</i>	mass to charge ratio
NAA	neutron activation analysis
NMR	nuclear magnetic resonance
Pro-ala	proline-alanine
Pro-gly	proline-glycine
Py-GC/MS	pyrolysis-gas chromatography/mass spectrometry
TAG	triacylglycerol
TD-GC/MS	thermal desorption-gas chromatography/mass spectrometry
TLC	thin layer chromatography
TLE	total lipid extract
TMCS	trimethylchlorosilane
TMS	trimethylsilyl

This text utilises both IUPAC and trivial nomenclature depending upon the subject matter and context of discussion. Wherever possible the system that facilitates greatest ease of understanding and/or consistency of the subject under discussion has been adopted.

PUBLICATIONS ARISING FROM THIS WORK

Buckley, S.A. & Evershed, R.P.

- 2002, Chemical Investigation of Horemkenesi, *Horemkenesi: May He Live Forever!* (ed. M. Ponsford) (in press)
- 2001, The Organic Chemistry of Embalming Agents in Pharaonic and Graeco-Roman Mummies, *Nature* (forthcoming)

Buckley, S.A., Stott, A.W. & Evershed, R.P.

- 1999, Studies of organic residues from ancient Egyptian mummies using high temperature-gas chromatography-mass spectrometry and sequential thermal desorption-gas chromatography-mass spectrometry and pyrolysis-gas chromatography-mass spectrometry, *The Analyst* 124, p.443-452

ANCIENT EGYPTIAN CHRONOLOGY

PREDYNASTIC PERIOD – 5500-3100 BC

Badarian Period – 5500-4000 BC

Amratian (Nagada I) Period – 4000-3500 BC

Gerzean (Nagada II) Period – 3500-3100 BC

EARLY DYNASTIC (PROTODYNASTIC/ARCHAIC) PERIOD – 3100-2686 BC

Ist dynasty – 3100-2890 BC

IInd dynasty – 2890-2686 BC

OLD KINGDOM – 2686-2181 BC

IIIrd dynasty – 2686-2613 BC

IVth dynasty – 2613-2494 BC

Vth dynasty – 2494-2345 BC

VIth dynasty – 2345-2181 BC

FIRST INTERMEDIATE PERIOD – 2181-1985 BC

VIIth dynasty – 2181-2125 BC

VIIIth dynasty – 2181-2125 BC

IXth dynasty – 2160-2025 BC

Xth dynasty – 2160-2025 BC

XIth dynasty – 2125-1985 BC

MIDDLE KINGDOM – 1985-1650 BC

XIIth dynasty – 1985-1795 BC

XIIIth dynasty – 1795-1650 BC

XIVth dynasty – 1750-1650 BC

SECOND INTERMEDIATE PERIOD – 1650-1550 BC

XVth dynasty – 1650-1550 BC

XVIth dynasty – 1650-1550 BC

XVIIth dynasty – 1650-1550 BC

NEW KINGDOM – 1550-1069 BC

XVIIIth dynasty – 1550-1295 BC

XIXth dynasty – 1295-1186 BC

XXth dynasty – 1186-1069 BC

THIRD INTERMEDIATE PERIOD – 1069-747 BC

XXIst dynasty – 1069-945 BC

XXIInd dynasty – 945-715 BC

XXIIIrd dynasty – 818-715 BC

XXIVth dynasty – 727-715 BC

LATE PERIOD – 747-332 BC

XXVth (Kushite) dynasty – 747-656 BC

XXVIth (Saite) dynasty – 664-525 BC

XXVIIth (First Persian Period) dynasty – 525-404 BC

XXVIIIth dynasty – 404-399 BC

XXIXth dynasty – 399-380 BC
XXXth dynasty – 380-343 BC

SECOND PERSIAN PERIOD – 343-332 BC

PTOLEMAIC PERIOD – 332-30 BC

ROMAN PERIOD – 30 BC-AD 395

COPTIC (CHRISTIAN) PERIOD – AD 395-642

ISLAMIC PERIOD – AD 642>

(nb. All dates before 690 BC are approximate and several of the dynasties run simultaneously)



KEY

- | | |
|----------------------------------|--------------------|
| 1 OK male adult | 10 GR female adult |
| 2 MK male adult ('Khnumnakht') | 11 GR male adult |
| 3 2IP female adult | 12 GR male adult |
| 4 2IP child | 13 GR male child |
| 5 NK (?) adult | 14 GR male child |
| 6 3IP male adult ('Horemkenesi') | 15 3IP hawk |
| 7 3IP female(?) adult | 16 3IP hawk |
| 8 3IP female adult ('Neskhons') | 17 LP cat |
| 9 LP male adult ('Pedeamun') | 18 LP ibis |

Map of Egypt showing provenance of mummies studied in this thesis

CHAPTER 1

Introduction

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1.1 GENERAL INTRODUCTION

Ancient Egyptian mummification has been a source of fascination for over two millennia. It was an integral part of a culture which required the preservation of the body to pass successfully on to the afterlife. Yet the practice did not reflect a morbid preoccupation with death, and was simply the means by which the pleasures of life could be extended into eternity.

The Egyptians' so-called 'art' of mummification developed - and regressed - over a four millennia span, the technology employed changing and evolving as a result of observation, experimentation and simple trial and error. Ritual, cultural and social influences also played parts in the process, and these changed as a result of the various influences to which Egypt was exposed. The development of funerary practices was also greatly affected by topography and geography. Dependent upon the River Nile as their sole source of water, the Egyptians used its fertile banks for agriculture close to which they built their houses, with their dead buried some distance away at the edges of the desert (ancient term 'deshret') which makes up 96% of the country's total land mass (Fig. 1.1).

1.2 ARCHAEOLOGICAL EVIDENCE FOR MUMMIFICATION: CHRONOLOGICAL DEVELOPMENT

1.2.1 Human mummification

Throughout ancient Egyptian history the vast majority of individuals were buried on their side in the flexed position in shallow pits in the sand, and surrounded by their personal belongings for use in the afterlife; this was the standard method of burial for all during the Predynastic Period (5500-3100 BC) (Smith & Dawson 1924, p.72-73, Fig.2; Reisner 1908; Cockburn et al. 1998, p.17; Lucas 1989, p.270; Andrews 1990, p.5, Fig.1; Harris & Wente 1980, Fig.1.6). Direct contact with hot sand served to rapidly and thoroughly desiccate the body, and in the absence of moisture necessary for bacteria to function, the soft tissue, hair and nails is generally very well preserved (Cockburn et al. 1998, p.17; Dunand & Lichtenberg 1994).

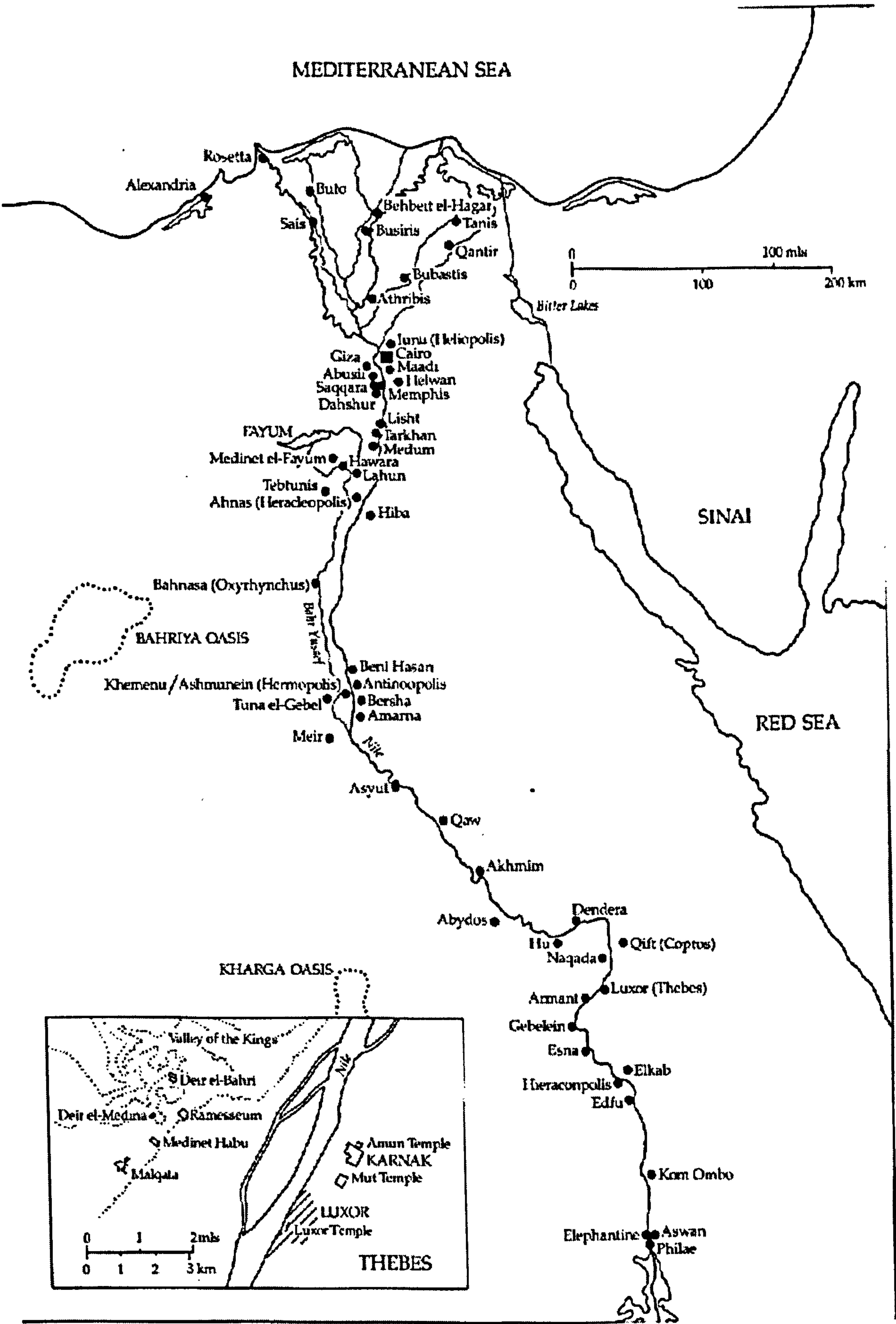


Fig.1.1 Map of Egypt

Yet their close proximity to the surface of the sand left the bodies vulnerable to the predations of both animals and grave robbers. This led to an adoption of protective measures, which in conjunction with ritual consideration and a desire to demonstrate the increasing status of certain members of society, resulted in the construction of purpose-built burial structures (mud-brick ‘mastabas’ and rock-cut tombs) for the elite. Yet no longer in close proximity to the desiccating sand, the body rapidly decomposed (Andrews 1984, Fig.41), and so it became necessary to produce an alternative means of preserving the body using artificial methods.

The earliest evidence yet found for attempts to artificially preserve the dead has been discovered within the last three years at the workers’ cemetery at Hierakonpolis (Kom el-Ahmar) in southern (‘Upper’) Egypt (personal communication, J. Fletcher) (Ill.1). Excavations at the site are continuing to reveal traces of ‘resin’-impregnated reed matting wrapped around bodies dated by the accompanying pottery vessels to c.3400 BC. This date would place the beginnings of Egyptian mummification 600 years earlier than previously thought and would have significant implications for the current understanding of the culture. Yet it must be stated that at this stage the evidence is based solely on visual observations, which cannot be used to confirm a ‘resin’ origin since the decomposition of body fats, etc. can produce a material with a resin-like appearance (Buckley et al. 1999, p.443-452).

Prior to this potential discovery, the earliest evidence for artificial preservation was linen-wrapped bodies of the Early Dynastic Period (3100-2686 BC). Excavations at the Ist dynasty tomb site of King Djer at Abydos revealed human remains in the form of a skull and part of a forearm, although the extent to which preservative materials were used cannot be ascertained, since the skull was lost and the arm, stripped of its jewellery, was thrown away (Petrie 1901, p.16, pl.1). ‘Resin’ impregnated linen was supposedly used widely during the Early Dynastic Period and early Old Kingdom (2686-2494 BC), the linen moulded to the contours of the face and body in order to give a life-like appearance (Englebach & Derry 1942, p.241). The body of an adult female was discovered in a IInd dynasty burial at Sakkara wrapped in 16 layers of linen (Quibell 1923, p.11, 19, 28, 32, pl.XXIX.3; Smith & Dawson 1924, Fig.1) (Ill.2), the suggestion being made that the corroded nature of the inner layers closest to the body most likely indicated the use of crude natron (sodium salts) as a preservative, although there was no scientific evidence to support this (Smith & Dawson 1924, p.74). Despite the use of linen, either that described as ‘resin’-

impregnated or otherwise, it must be stated that there was little meaningful attempt at mummification at this time. Such bodies are poorly preserved, and with very little soft tissue remaining they are largely skeletal.

The practice of moulding linen around the body continued into the Old Kingdom (2686-2181 BC), with the remains of a single foot with individual toes carefully wrapped, together with fragments of spine and pelvis (Englebach & Derry 1942, p.243) discovered in the IIIrd dynasty burial chamber of King Djoser within his Step Pyramid at Sakkara (the remains are those of several individuals recently dated to both the Old Kingdom and Saite/Ptolemaic Periods, Strouhal et al. 1998). Contemporary remains recovered from the necropolis of Beni Hassan were also wrapped in linen, although again there had been no significant attempts to mummify the bodies (Garstang 1907, p.30). However, by the IVth dynasty (2613-2494 BC) there is firm evidence for the use of desiccants and evisceration, both of which are crucial for the optimum preservation of the body and as such are key elements in the classic mummification process. The practice of evisceration also marks a change in the laying out of the body, the former flexed position now extended to give full access to the abdominal region, a change which is also reflected in the elongation of coffin size. The first positive evidence for evisceration comes from the intact Giza burial of Queen Hetepheres (c.2600BC) and despite the mysterious absence of the queen's body, her viscera were discovered inside her calcite 'canopic' chest in a liquid solution of 3% natron. This is also the first positive evidence for the use of a desiccant within a funerary context (Reisner 1942, p.155-156; Andrews 1984, Fig.5), the 272 days taken for mummification at this time almost certainly a direct result of using liquid natron rather than its more efficient dry form employed later (Harris & Wente 1980, p.9-10, discussed below).

In a contemporary burial at Medum, the mummy of the nobleman Ranefer was found to be wrapped in multiple layers of linen wrappings, the outer of which were saturated with "*resinous paste*" (Smith & Dawson 1924, p.74-75, Fig.3; Englebach & Derry 1942, p.241; Cockburn et al. 1998, p.24-25). The care taken in moulding the linen to the body was such that evidence for circumcision could be observed, in addition to which the eyes, brows and moustache were painted in detail. Although the brain remained *in situ*, the internal organs had been removed and the internal thoracic cavity packed with "*resin-soaked linen*" (Smith & Dawson 1924, p.74-75), although the mummy's subsequent destruction in wartime bombing prevents further analysis. A contemporary burial from Giza contained the mummy of an unnamed woman:

“very carefully wrapped, the fingers and toes individually wrapped...and the body and upper legs wrapped in thirty-seven layers of linen bands into which were inserted wads and pads of linen to give a more life-like appearance. The breasts were made of layers of narrow bandages... molded using wet resin. The head and face were similarly sculpted” (Boston 1988, p.77).

The Vth dynasty (2494-2345 BC) male mummy of Setka found at Giza had also been eviscerated and the body cavity packed with linen, although the soft tissues had largely decomposed to leave the linen wrappings in direct contact with the bones. The inner wrappings were also said to have been ‘burnt’ by the body fluids (Englebach & Derry 1942, p.241, 244-245). The contemporary mummy of ‘chief of weavers’ Watay (though generally erroneously referred to as ‘Nefer’ after the owner of the Sakkara tomb it was discovered in) was again wrapped in carefully moulded linen soaked in “*an adhesive*” (Cockburn et al. 1998, p.25; Harris & Weeks 1973, p.17, 83) (as opposed to the equally vague and dubious term ‘resin’), with a coating of stucco plaster then applied (Boston 1988, p.91-92) and the facial features painted on (Cockburn et al. 1998, p.25; Harris & Weeks 1973, p.83). The same treatment had also been used in the mummification of an unnamed VIth dynasty body found at Giza, the subsequent examination revealing that evisceration had not occurred (Englebach & Derry 1942, p.242; Harris & Wente 1980, Fig.1.11).

Evidence for true mummification therefore first appears with the use of desiccants and evisceration during the IVth to VIth dynasties of the Old Kingdom. Yet the brain was not generally removed, and the soft tissue was poorly preserved following the modelling of the features in attempts to obtain a life-like appearance, necessary so the deceased’s spirit could recognise the body, reunite with it and so pass into the afterlife (Faulkner 1985). Developments in mummification ran broadly parallel with political developments, and at the height of the Old Kingdom it was a process restricted to royalty, who could extend it to their officials as a privilege, accompanied by burial in the royal cemeteries of Sakkara, Medum or Giza. With the subsequent decline of royal power at the close of the Old Kingdom, decentralisation during the First Intermediate Period (2180-1985 BC) led to such officials moving back to their own localities where they were now buried, rather than close to their king. This explains the increasingly wide distribution of mummified remains discovered throughout the Nile Valley. One VIIth dynasty (c.2181 BC-2125 BC) tomb at el-Hagarsa in Middle Egypt contained six mummies, each wrapped in linen, and although three had cartonnage coverings on the face and chest areas there was no evidence for any other preservative procedures (Kanawati 1993, p.36). This was also the case in the burials of the elderly woman Merirtyef at Mesheikh (Boston 1988, p.103-104) and the IXth dynasty

official Pepiseneb from Sheikh Farag, his heavily wrapped mummy examined by CT scan revealing “*no evidence of the removal of the brain*” (Boston 1988, p.106).

Following a period of civil war, the rise of the powerful XIth dynasty (2125 BC-1985 BC) in Thebes produced a concentration of burials in the area of Deir el-Bahari associated with the court of King Montuhotep II (2055 BC-2004 BC). Chief among these were the mummies of his wives Henhenet, Kemsit, Kawit and Sadeh and ‘great royal wife’ Ashayet (Ill.3), and although all have suffered appallingly since, their mummies were found largely intact. Examination of Ashayet’s remains revealed that the brain remained *in situ*, and whilst there were no signs of an embalming incision the dilated state of the vagina and rectum suggested that “*cedar oil or oleo-resin*” may have been injected into the body to preserve the internal organs which were found intact, albeit in a shrunken state (Englebach & Derry 1942, p.248). Shiny, black particles found on the inner surface of the rectum were identified as resin by Lucas (Englebach & Derry 1942, p.248), although more recent work suggests that the particles simply represent the natural degradation of body fats (adipocere) and are not resin (personal communication, R. Walker). Although initial studies suggested that Ashayet and Henhenet (Smith & Dawson 1924, Fig.6) had both undergone similar treatment and that “*some form of resin had been applied with the bandages next to the body*” (Englebach & Derry 1942, p.248), the previous ‘identification’ of the shiny particles as resin casts doubt on these early conclusions. Yet recent studies have confirmed that Ashayet’s naturally dark brown hair had been enhanced with a tannin-based vegetable colorant, her mid-length plaits prepared for burial with an application of a ‘resin’ fixative, and the epidermis remaining on her hands and feet stained orange-red with henna (*Lawsonia inermis*) (Fletcher, forthcoming). The mummies of three further women associated with Montuhotep II’s court were also found at the site, and all extensively tattooed, the mummy of the priestess Amunet had been prepared in a similar way to that of Ashayet given its significantly dilated vagina (Englebach & Derry 1942, p.249; Harris & Weeks 1973, p.17; Harris & Wente 1980, p.13, Fig.12.a,b). None had been eviscerated and in each case the brain remained intact (Harris & Wente 1980, p.13), the further discovery of the materials used to embalm them revealing the practice of burying everything connected with the deceased for ritualistic reasons.

The largest group of remains associated with the site are the so-called ‘slain soldiers’, sixty male bodies placed in a mass burial close to the tomb of their king Montuhotep II. Given their location and the nature of their wounds (caused by weapons common in northern

Egypt at that time), the men are believed to have died during the civil war conflicts. Close examination of their remains also suggests that they had lain on the battlefield for some time and their bodies picked at by birds, before being gathered together and wrapped in royal linen for a state burial (Winlock 1945). Another intact Theban burial of the period revealed the superbly mummified body of the estate manager Wah. Beneath copious external wrappings of 375 square metres, “*a layer of dark resin*” (Hayes I 1959, p.303) overlaid further linen wrappings, beneath which was more so-called ‘resin’. Partial evisceration indicates the resumption of true mummification after a 300-year hiatus, and although the contemporary mummy of the Vizier Ipi had been destroyed in antiquity, his ransacked Theban tomb still contained the embalming equipment originally used (Hayes I 1959, p.165). Of similar XIth dynasty date, CT scans of the linen-moulded mummified head of the official Djehutynakht revealed:

“one rather surprising find: the brain had been removed, a process not performed with regularity until Dynasty 18. The head shows that the process was in an experimental stage, because the route of removal was very different from that in use during later times, namely through the nose. Holes were punched through Djehuty-nakht’s maxillary sinuses, and the brain was removed through the ethmoid air cells and sphenoid sinuses. This process damaged the skull, resulting in fractures in the outer orbits of the eyes” (Boston 1988, Fig.6, p.16, 112).

Despite the extensive damage to his Bersheh tomb, the remains of his canopic equipment still with traces of the original linen-wrapped contents attest that evisceration had again taken place (Boston 1988, p.111; Serpico & White 1998 p.1037), and a loose piece of linen from the burial “*was saturated with a gummy substance, darkening the colour to a red-brown*” (Boston 1988, p.117).

By the beginning of the Middle Kingdom (1850-1650 BC) there was a clear ‘democratisation’ of burial practices. Courtiers’ coffins were frequently inscribed with the funerary texts once restricted to royal use, and as more people are provided with mummification and the necessary ritual accompaniments, the cult of the god Osiris flourished accordingly. As god of resurrection, Osiris was the first individual to be mummified when his sister-wife Isis restored his faculties to conceive their child. Thereafter, deceased individuals were identified with the god in order that their soul complete the hazardous journey to immortality, for which their body, as home of their soul, was a vital component.

The Middle Kingdom monarchy relocated north back to the traditional capital Memphis, and no less powerful than their Old Kingdom predecessors were buried in pyramids at the traditional burial ground of Dahshur and at Hawara, Lisht and Lahun. Yet the damper

conditions of these northern cemeteries resulted in significantly greater decomposition, as exemplified by the total disintegration of Amenemhat III's daughter Neferuptah in her intact yet completely waterlogged burial at Hawara (Farag & Iskander 1971, p.22-23, 27, pl.XX). Of the few royal remains to have survived, the Dahshur tomb of Sesostris III's daughter Menet contained "*resin-treated bones*", the remains of the XIIIth dynasty King Hor (c.1795 BC) again found at Dahshur revealing evidence of both natron and apparent 'resin' treatment. The semi-intact burial of Senebtisi at Lisht from the early XIIth dynasty still contained her poorly-preserved mummy:

"...wrapped in alternating layers of sheets and bandages and covered over the front with a coating of resin, evidently poured on in liquid form....The viscera had been extracted through an incision over the left groin and the body cavity packed with sawdust and wads of linen soaked in resin. The heart wrapped in linen had been replaced in the body but the liver, intestines and two other organs had been permanently removed and placed in the four canopic or visceral jars" (Hayes I 1959, p.305; Mace & Winlock 1916, pl.Xa),

into which had then been poured "*molten resin*" (Smith & Dawson, p.81). It was also noted that the embalming incision had been plugged with further 'resin'-soaked linen, with the brain left *in situ* and the eyes also covered with yet more 'resin' (Smith & Dawson 1924, p.81; Mace & Winlock 1916). The mummies of several XIIth dynasty male officials have also been discovered in somewhat better condition. In the case of Karenen found at Sakkara, his brain remained *in situ* whilst evisceration had occurred through the embalming wound in the left flank. With the exception of the heart, the contents of both the abdominal and thoracic cavities had been removed, and the interior then filled with "*bundles of linen, on some of which incrustations of resin were clearly discernable*". The body was wrapped in more 'resin' soaked linen and the face "*thickly smeared with resin, plugs of which were also placed in the nostrils. The eye-sockets were filled with plugs of linen*" (Smith & Dawson 1924, p.80-81). The mummies of the 'Two Brothers' Khnumnakht and Nekhtankh were found in their joint burial further south at Rifeh and retained a limited amount of soft tissue. Although the brain again remained *in situ*, evisceration had been carried out and the thoracic cavity packed with matting or coarse linen (Murray 1910; Smith & Dawson 1924, p.82).

In general, Middle Kingdom mummification was of quite a high standard. After the internal organs were removed and the lungs, stomach, liver and intestines placed in the appropriate canopic jar, the thoracic cavity was generally filled with linen, sometimes sawdust, impregnated with 'resin'. The body was then desiccated under piles of dry natron and once the moisture had been removed the external surfaces were coated in 'resin' to prevent re-hydration.

Following the decline of the Middle Kingdom under a long series of ephemeral rulers, Palestinian settlers took advantage of Egypt's internal instability to take the throne as the 'Hyksos' ('Rulers of Foreign Lands') of dynasties XV and XVI. This marked the beginning of a second Intermediate Period (1650-1550 BC) as the country once again divided between the northern Hyksos and their southern native rivals based at Thebes and a century of intermittent civil war followed. In c.1550 BC the Thebans drove out the Hyksos to take Egypt back, as once again the political situation can be seen to directly affect the development of mummification and the nature of funerary practices. With Thebes again the capital of Egypt's growing empire, the desert cliffs on the opposite banks of the River Nile became the royal burial ground for the next 500 years, the optimum nature of its arid conditions combined with increasingly sophisticated preservation techniques producing superb results, as exemplified by the large number of royal mummies from the Theban necropolis.

Due to the extent of tomb robbing in antiquity, the royal mummies of the New Kingdom had been salvaged and rewrapped by priests and repeatedly moved (Buckley & Rose 2000; Fletcher 2000b, p.3) before being finally buried in two great caches discovered intact in the late 19th century. In 1881 the Deir el-Bahari tomb of Pinudjem II (DB.320) was found to contain 50 mummies including 13 kings, buried in 934 BC, whilst in 1898 the tomb of Amenhotep II (KV35) in the Valley of the Kings revealed a further 11 kings (Reeves & Wilkinson 1996, p.194-195, 198-199). Taken to Cairo Museum and examined here shortly after their arrival in the only comprehensive study to date (Smith 1912), these early findings of fifty royal mummies are nevertheless vital to the understanding of mummification practices of the time, especially given the rapid deterioration of these mummies since their discovery. Subsequently there has only been one other study, which concentrated almost exclusively on radiographic examination (see below, also Harris & Weekes 1973; Harris & Wente 1980).

The earliest of these royal mummies is that of the XVIIth dynasty king Seqenre II, the Theban warlord killed leading the final push against the Hyksos. His mummy is probably the most dramatic ever found, with lips drawn back in a grimace and his head repeatedly punctured by Hyksos weaponry, including a dagger behind the ear, a mace into the cheekbone and an axe blow to the forehead. In the initial examination, it was found that:

"All that now remains ... is a badly damaged, disarticulated skeleton enclosed in an imperfect sheet of soft, moist, flexible, dark brown skin, which has a strongly aromatic, spicy odour. The skin resembles that of mummies of the Coptic [Christian] Period after they have been exposed to the air and the

preservative salts have deliquesced and softened the tissues... [We have] been unable to find in Saqnounri's [Sequenre] skin any greater quantity of chloride of sodium than occurs in untreated human tissues. The spicy odour of the skin is due to the fact that it has been sprinkled with powdered aromatic wood (or sawdust)" (Smith 1912, p.1).

These observations were then followed up by the 'chemical examination' of a piece of Sequenre's skin at Cairo Medical School, but tests were "unable to find any excessive quantity of salt in it, in fact no greater quantity of sodium chloride than the normal tissues of the human body contain" (Smith 1912, p.9, pl.II-III). In their visual inspection, more recent investigators were only able to add that the brain had been removed and there were no packing materials (Harris & Wente 1980, p.169).

Although the mummy of Sequenre's eldest son Kamose fell to dust at its discovery, that of Kamose's wife Tetisheri (Smith's 'Unknown Woman B') did survive and is that of an 'elderly adult female' whose "skin is blackened.....and the face is coated with a black, shining resin-like material, to which the bandages are adhering" (Smith 1912, p.14, pl.IX-X). Sequenre's youngest son Ahmose became the first king of the XVIIIth dynasty and New Kingdom, the time when mummification achieved its greatest results. Although Ahmose's mummy had suffered at the hands of ancient plunderers:

"It would, no doubt, have shown more evidence of this rough treatment if it were not for its strength and stony hardness, as the result of the application of an abundant layer of black resinous paste to the surface of the whole body. The head, both face and scalp, are still thickly encrusted with this black material. ...With the head encrusted with a thick layer of material of stony hardness it is not possible to obtain accurate cranial measurements" (Smith 1912, p.16, pl.XI-XII).

The examiners go on to comment that:

"the cranial cavity is tightly packed with linen right down to the foramen magnum; and it seems incredible that this could have been accomplished through such a narrow cleft as either of the nasal fossae of this mummy, without damaging the septum. Moreover, there is the curious and significant fact that the atlas is missing; and the upper surface of the axis and the neighbouring part of the occipital bone are thickly coated with a mass of black, shining material (?resin), which must have been applied directly to the surfaces of the bones. This raises the possibility...that through an incision upon the left side of the neck the atlas was excised and the brain removed through the foramen magnum, which would be exposed by such a operation....The body, like the head, was smeared with a thick layer of black paste, which prevents an examination of the embalming wound. The perineum was covered with a thick mass of resinous material; and the external genitalia were also coated with the same substance." (Smith 1912, p.17-18).

The mummy of Ahmose's sister-wife Queen Ahmose-Nofretari has also survived, the wrappings which:

"still adhere to the mummy are of the dark yellowish and reddish-brown colours (as the result of being impregnated with a resinous solution) usually found in bandages swathing XVIIIth dynasty mummies. The skin is blackened like most of the mummies of this period. As in most of the mummies of the first half of the XVIIIth dynasty the perineum is coated with a thick plate of solidified resinous paste. The embalming wound... is plugged with linen smeared with black resinous paste, which bears the impression of the leaf-like plate which once covered it. The cranium is of a broad, sphenoid form.....even if some allowance be made for the resin-encrusted scalp" (Smith 1912, p.13-14, pl.VII).

The visual inspection of the later investigators found no traces of internal packing material, and with the nasal septum intact the absence of the brain indicates it must have been removed via an alternative route, presumably at the base of the skull (Harris & Wente 1980, p.168-169). The mummies of several other royal women from this period show similar features. In the case of an elderly adult female possibly to be identified as Ahmose's daughter Meritamun:

"the embalming wound has been made in the usual position on the left flank and the body cavity was packed with pads of linen soaked in a solution of resin in the manner customary in the times of the XVIIIth dynasty. The pelvis is packed with a had mass of resin and aromatic sawdust and a small quantity of similar material is smeared over the perineum, but not in sufficient quantity to hide the rima pudenda, as was the custom in the middle and later periods of the XVIIIth dynasty. The body was enveloped in large quantities of linen soaked in a solution of resin, which is peculiarly distinctive of the XVIIIth dynasty" (Smith 1912, p.7).

The mummy of Lady Anhapu again has:

"the usual incision made in the left flank and parts of the contents of the body cavity removed. Salt was then applied, in all probability, to the surface of the skin of the whole body; and after a time the excess of salt was removed and aromatic powdered wood was sprinkled over the whole body, which was then wrapped in large quantities of linen saturated with a solution of resin. The hair ...was plaited and thickly smeared with a paste, apparently of a resinous material" (Smith 1912, p.10-11, pl.IV-V).

The wrappings of Lady Rai's mummy:

"had been soaked in a solution of resin in the manner distinctive of the early part of the XVIIIth dynasty. The body and face were covered with a thin layer of a mixture of sand and powdered resin... The embalming-wound....was stuffed with a plug of fine linen, freely sprinkled with a mixture of sand and powdered resin; and there is the impression of a fusiform plate, which was placed over the embalming-wound. This form of plate is distinctive of the XVIIIth dynasty" (Smith 1912, p.11-12, pl.VI, Fig.2).

Hontimihou's mummy had likewise:

"been wrapped in an enormous quantity of bandages saturated with a solution of resin. The body is packed with resin-saturated pads of linen" (Smith 1912, p.19, pl.XIV).

In the preparation of the mummy of Sitkamose "black resinous paste" had been:

"thickly smeared over what remains of the chest wall (and in fact over the whole body), there are very sharply defined impressions of the pectoral ornament...The whole body, including the face, is thickly smeared with a black resinous paste, in which fine bandages are embedded. It is, perhaps, characteristic of the XVIIIth dynasty embalming to cover the perineum with a large cake of black resinous paste, as in the case of Sitkamos[e's]' mummy. This practice began with Ahmosis [Ahmose]. The whole body was tightly packed with linen, some of it soaked in a solution of resin" (Smith 1912, p.21-22, pl.XVIII).

Later 'visual inspection' revealed the brain had been removed and there was no evidence for internal packing materials (Harris & Wente 1980, p.169). The mummy of King Amenhotep I has never been unwrapped, *"the wrappings...in such perfect condition that {it was} decided to let it remain untouched"* (Smith 1912, p.18, pl.XIII; Harris & Weeks 1973,

p.18). That of his successor Tuthmosis I (Harris & Weeks 1973, p.17) had originally been wrapped in “*resin-impregnated bandages*” which:

“can leave us in no doubt that the body was embalmed at some time during the XVIIIth dynasty. The excellent state of preservation of the body and the firmness and durability of the skin and tissues, indicate the attainment of perfection in the art of embalming unknown before the time of Ahmosis [Ahmose] I; and the fact that this result has been attained without plastering the body with a thick layer of resinous paste may be regarded as evidence that the mummification was done at a period subsequent to that of Ahmosis [Ahmose]. The rounding of the margins of the external auditory meatus makes it certain that the ear must have been plugged in the same manner as that of Thoutmosis [Tuthmosis] II, where a ball of resinous material is still in situ” (Smith 1912, p.25, 27, pl.XX-XXII).

This feature was also observed in the mummy of his son and successor Tuthmosis II, with:

“each external auditory meatus occupied by a round plug of resin. Both nostrils are distended with plugs of linen impregnated with resinous material” (Smith 1912, p.30, pl.XXIII-XXIV).

The later radiographic examination of the first two Tuthmosis kings revealed “*an intact nasal septum and the remains of the desiccated brain*” in both cases, with packing also used in the mouth of Tuthmosis II (Harris & Wente 1980, p.168-170). Although the mummy of the female pharaoh Hatshepsut has not been conclusively identified (Reeves & Wilkinson 1996, p.187), detailed information regarding mummification techniques during this reign comes from the Theban burial of the family of the great royal official Senmut (Lansing & Hayes 1937, p.4-39; Derry 1942, p.260-261; personal communication, R. Walker). Studies of the mummification techniques used on the bodies have shown that they were not all buried at the same time, the mummies of Senmut’s father, two female relatives and their three children little more than disjointed skeletons mixed with large quantities of sand wrapped in linen, whereas Senmut’s elderly mother Hatnefer had received a wealthy burial. Her carefully mummified body was described as “*well preserved*” after its examination *in situ* (Lansing & Hayes 1937, p.26), with further examination in Cairo revealing that the brain was still present and that no resin had been used during the mummification process, “*the abdomen and chest filled with tightly-rolled balls of linen*” only (Derry 1942, p.260). In recent re-examination her nails were found to be stained with henna, and her false hair treated with a ‘resin-like’ mixture (Fletcher, forthcoming). The differing levels of mummification amongst the family reflect the way in which Hatnefer outlived the others to enjoy the wealth brought by her son’s success, and when he buried his mother he had the rest of the family re-wrapped and re-interred with her.

The burial of the contemporary courtier Harmose ‘the Singer’, was discovered close by, his lute and a pot of yellow ‘resin’ found with his mummy. The surviving soft tissue included part of his scalp with quite long, bright blond hair, set in artificially-created curls fixed in

place with some form of resin, perhaps from the jar interred with him. Following recent microscopic analysis this appeared as *“a cracked golden coating resembling some sort of gum”* (Walton Rogers in Fletcher, forthcoming). This enhanced hair colour likely had ritual significance as a way to increase the deceased’s affinity with Hathor, goddess of music who was also known as ‘the Golden One’.

In the five year period between the preliminary unwrapping of Tuthmosis III’s mummy in 1881 and its re-examination in 1886 it was said that the body was coated with:

“a layer of whitish natron charged with human fat, greasy to the touch, foetid and strongly caustic”. It was suggested that “the whitish material in question was an efflorescence of fatty acids. Happily the face, which had been plastered over with pitch (resin) at the time of embalming, did not suffer at all from this rough treatment, and appeared intact when the protecting mask was removed. ...Through it the cavity of the abdomen can be seen to be packed with a quantity of cloth impregnated with resinous material. Both nares are stuffed with a black resinous mass” (Smith 1912, p.32, 34, pl.XXVIII; Fletcher 2000b.p.3).

The later radiographical examination revealed an intact nasal septum and the remains of the king’s desiccated brain (Harris & Wente 1980, p.168-169). The mummy of his son and successor Amenhotep II was studied using photographs in which:

“several pieces of resin-soaked linen will be seen adhering to the nose and mouth and hiding the features of the face. In the resin covering the fifth dorsal spine there is the distinct impression of a series of beads arranged in the pattern of the well-known pectoral ornament. On the back of the sacrum there was a geometrical pattern impressed on the resin. The pudenda were pushed in against the perineum and embedded in a great mass of resinous paste, which was spread over the whole perineal region” (Smith 1912, p.37-38).

Subsequent radiographical examination revealed the removal of the brain and its replacement with cranial packing (Harris & Wente 1980, 169). The abdominal cavity of the mummy of Tuthmosis IV had also been tightly packed through an excessively large embalming incision (Harris & Weeks 1973, p.40) using *“cloth saturated with resinous material, which formed a very hard, solid mass”*. The surface of his shoulder-length hair *“was studded with masses of dark foreign matter (embalming material)”*, and his mouth had been covered with a *“quantity of resinous material which covered the teeth”* (Smith 1912, p.43-45, pl.XXIX-XXX). Later radiographs revealed that this material had been used over oral packing, and the king’s nasal septa were intact (Harris & Wente 1980, p.169-170).

Three unidentified mummies discovered in a side chamber of the second royal cache were dated by the way in which certain embalming materials had been applied, particularly the large masses of ‘resin’ applied to the perineum. In the case of the ‘Elder Lady’, subsequently identified on the basis of hair analysis as Amenhotep III’s queen Tiye (Harris et al. 1978, p.1149-1151):

“the perineum was thickly plastered with resin in the form of a large cake. The rima pudenda is widely open and stuffed with linen from the inside (from the pelvis). The resinous paste was applied to the surface of this linen plug and to the skin areas surrounding it” (Smith 1912, p.39, pl.XCVII).

The abdominal cavity of the shaven-headed ‘Younger Woman’ found with her was:

“stuffed with balls of linen soaked in a solution of resin. As [before] a large mass of resin was spread over the whole perineum. Both in this mummy and in the other woman the rima pudenda was widely open and plugged from the inside with linen, on the surface of which the perineal mass of resin was smeared. Only the abdominal cavity was packed with linen (soaked in resinous solution), but great buttons of linen projected upwards into the thorax through the apertures in the diaphragm” (Smith 1912, p.41, pl.XCIX).

The third of the mummies from the side chamber was that of a young boy:

“the mass of brain and resin in the cranial cavity forming a perfect mould of the occipital fossae and the groove for the superior longitudinal sinus turning into the right lateral sinus. The material obtained from the cranial cavity has been analysed” (Smith 1912, p.41, pl.XCVIII),

although it proved *“impossible to identify: large amount of combined alkali present”* [sic] (Lucas 1908, p.143). If the identification of the ‘Elder Lady’ as Tiye is correct, it is more than likely that the younger woman and boy buried with her are members of the same family and relatives of Amenhotep III (personal communication, J. Fletcher). The mummies of the king’s own parents-in-law and Tuya have also survived and are without doubt the most technically perfect examples to have survived from the whole of ancient Egypt (Quibell 1908; Harris & Weeks 1973, p.19) (Ill.5), both of them eviscerated and Yuya’s embalming wound covered with a fine gold plate (Harris & Weeks 1973, p.19). Both exhibit startlingly yellow hair, and although it has been suggested that this is either evidence for their foreign origin, or the result of the embalming fluids acting on white hair (Smith in Quibell 1908, p.69), it is clear that dyes were also used during life to give much the same effect (Fletcher 2000, p.500).

Their life-like appearance is in sharp contrast to that of the king himself, one of the most damaged of all royal mummies, albeit one of the most intriguing. Initial examination of the body cavity revealed *“lumps of resin-impregnated linen”* together with bird bones and *“a human great toe and a left ulnar and radius”* which suggest hasty rewrapping of the mummy in antiquity.

“Although it was a great disappointment to find only these broken and blackened bones to represent the body of Amenhotep [Amenhotep] “the Magnificent”, the study of the remains revealed certain facts of singular interest to the student of the history of embalming. For the attempt had been made to restore to the limbs and body of the dead Pharaoh some semblance of the form these parts had possessed in life, but had lost during the earlier stages of the process of mummification. This was accomplished by stuffing under the skin of the legs, arms, neck and perhaps other parts of the body, a resinous mass, which was moulded into form; so that when it set, the members of the mummy consisted of masses of stony hardness with a covering of skin. Precisely how the packing material was inserted under the skin in the case of Amenhotep [Amenhotep] III is unknown; but if we study the analogous

process of packing that was revived three dynasties later we shall obtain very precise information as to how it was done in the times of the XXIst and XXIInd dynasties, and how it may have been done in the case of Amenhotep [Amenhotep] III. A sample of the packing taken from Amenhotep's [Amenhotep's] left arm was examined [and found to] consist of a resin mixed with 14.3 0/0 of inorganic matter, of which 7.5 0/0 consisted of a mixture of carbonate, sulphate and chloride of sodium, i.e. crude Egyptian 'natron'. Resinous material such as this is not known to have been employed at any other period for packing underneath the skin. In the time of the XXIst-XXIInd dynasties, linen, mud, sand, sawdust or cheese-like substances (mixtures of fat and soda) were the stuffing materials employed. In no mummies earlier than Amenhotep [Amenhotep] III is there any evidence to indicate, or even to suggest, that any such curious procedure was put into practice; and as I have examined the mummies, not only of Amenhotep's [Amenhotep's] immediate predecessors, but also of his wife's parents, Yuaa and Thuaa, without finding any trace of stuffing in the limbs, it is safe to conclude that this addition to the embalmers' technique was invented at or near the close of the reign of Amenhotep [Amenhotep] III, when the spirit of change was rife in Thebes, and the old conventions in the Arts, as well as in worship, were being overthrown. Whether or not the bodies of the XVIIIth dynasty successors of Amenhotep [Amenhotep] III were submitted to this strange process of packing it is now impossible to say, because nothing but skeletons of some of them have come down to us. But we do know that none of the Royal Mummies of the XIXth and XXth dynasties were so treated; and it was not until the close of the XXth or the beginning of the XXIst dynasties that the practice was revived, and became part of the regular routine of mummification during the XXIst and XXIInd dynasties. This stuffing of material under the skin must not be confused with the process of packing the cavity of the body, which always formed part of the process of embalming from the time of the Ancient Empire [Old Kingdom] until Roman times" (Smith 1912, p.49-50, pl.XXXII-XXXV).

Following chemical analysis at the time, samples of the resinous material taken from Amenhotep's cheek and left arm were reported to contain myrrh (Lucas 1908, p.142-143), with more recent radiographic examination revealing the use of packing in the mouth (Harris & Wente 1980, p.170). The early observations make particular sense in the light of recent epigraphic work which has revealed that Amenhotep III was unique in the way in which he chose to ritually transform himself into the living sun, his recently discovered statuary reinforcing his divine solar status whilst still a living king. The similarly unique way in which his skin had been imbued with the golden, reflective qualities of 'resins' were almost certainly linked with the king's choice of solar identity. The fact that the mummy is described as "*stony hardness with a covering of skin*" must also be part of the king's religious ideology in his desire to achieve a permanent statue-like appearance, particularly when the same embalmers were able to transform his contemporaries into far more 'life-like' mummies.

The largely skeletal remains identified as Amenhotep III's son and successor Akhenaten (Smith 1912, pl.XXXVI-XXXVII) reveal traces of 'resinous material' on the linen wrappings (personal communication, J. Fletcher), but the loss of almost all soft tissue makes it impossible to study the mummification techniques used. It can only be said that the missing nasal septa suggest that the brain had been removed (Harris & Wente 1980, p.168). Tutankhamen appears to have been mummified in the same manner as his grandfather Amenhotep III, whom he regarded as his predecessor following the obliteration of his father's memory for political reasons. Following the celebrated discovery of his intact tomb

in 1922, his mummy was unwrapped *in situ* exactly 3 years later, the stage of development of the flora used in the wreaths adorning the inner coffin suggesting a date of burial between mid-March and late April. It was estimated at the time that “*something like two bucketsful of the liquid unguent had been poured over the golden coffin, and a similar amount over the body inside*” (Carter 1927, p.87), such quantities effectively welding the body to the base of his innermost coffin. Once the body had been freed, and the sixteen layers of linen interspersed with juniper berries removed, it was noted that these same anointing materials described as “*fatty matter, resin, and possibly wood pitch*” had damaged both the coffins and the mummy alike.

“The action of the composite liquid employed has been threefold: first, the decomposition of the fatty matter, by producing fatty acids, has acted destructively upon certain qualities of the glass inlay and cement of the objects; secondly, the oxidation of the resin has given rise to a kind of slow spontaneous combustion, resulting in the carbonisation of the linen fabric and, in a less degree, of the tissues and even the bones of the mummy; thirdly, the quantity of liquid poured both over the innermost coffin and mummy itself was sufficient to form a pitch-like cement which consolidated the contents” (Carter 1927, p.101).

Nevertheless, an autopsy was finally carried out and it was noted that:

“the abdominal wall exhibited a marked bulging on the right side...due to the forcing of the packing material across the abdominal cavity from the left side where the embalming incision is situated... The lips of the wound are inverted owing to the forcible packing of the abdomen with a mass of linen and resin, now of rock-like hardness. The plugs filling the nostrils and the material laid over the eyes were of linen impregnated with resin. The skull cavity is empty save for some resinous material which had been introduced through the nose” (Derry in Carter 1927, p.222-227; Derry in Leek 1972).

Following a more recent radiographical examination (Harris & Wente 1980, Fig.1.13-14), a small fragment of detached bone was revealed inside the cranium, and despite being sensationalised as evidence of ‘foul play’, it is simply part of the ethmoid bone broken during removal of the brain via the nasal passages. The “*whitish spots on the skin over the upper part of the back and shoulders*” proved to be composed of common salt with a small admixture of sodium sulphate, “*in all probability derived from the natron used in the embalming process*” (Harris & Wente 1980, p.17).

The materials used in Tutankhamen’s mummification and funeral were buried separately, and included quantities of natron and linen and a miniature death mask (Winlock 1941). This seems to have originated from one of the two mummified female fetuses buried in his tomb, the first of five months gestation and the second between eight to nine months’ gestation. Whilst the younger had simply been wrapped in linen with “*no indication as to how the body was preserved*” (Derry in Carter 1933, p.167), the elder had been eviscerated:

“the abdominal wall ...opened by an incision 18mm in length on the left side immediately above the inguinal ligament and parallel to it. The opening was closed with a sealing of resin. The abdominal

cavity is stuffed with linen impregnated with some saline material" (Derry in Carter 1933, p.169; personal communication, J. Fletcher).

In chronological terms, the next royal mummy is that of Seti I, dating from the early XIXth dynasty (III.6). It was noted that:

"the whole surface of the body, excepting the head only, is covered with a black mass of bandage impregnated with resinous material. All the exposed areas of the skin, including the face, are quite black, but when the head was first exposed in 1886 the skin was distinctly brown and not black. The left side of the chest is stuffed with black masses of resin-impregnated linen, now of stony hardness. In the apex of the right side of the thorax there is a solid black mass about the size of a closed fist. It is not linen, but some brittle jet-black material, with a shining surface when fractured. Below this and separated from it by a wide interval there is a large heart-shaped mass of stony consistency, which has a dull brown colour when scraped. It seems to be a viscus, perhaps the heart, but it is not possible to express any positive opinion without cutting it and submitting a piece of it to microscopical examination. Packed around this mass, which is probably the heart, there is a considerable quantity of resin-impregnated linen. The abdominal cavity was partially filled with black masses of similarly treated cloth, but there is no trace of any viscus" (Smith 1912, p.57-58, pl.XXXVIII, XL, XLI; for black colour see Harris & Weeks 1973, p.18).

The mummy of his son and successor Ramses II has been subject to the most exhaustive studies undertaken on any of the royal mummies (Paris 1985), although initial studies were also quite extensive.

"The eyebrow is preserved only on the right side. Traces of a layer of dark brown or black paint still persist on the superciliary ridges. The skin of the forehead is of a light yellow colour, thickly spotted with reddish brown patches. This mummy reveals a distinct advance in the technique of the embalmer's art – for the first time it became possible to preserve the skin without the dark brown or black discolouration that occurred invariably in earlier attempts at mummification. The soft parts of the nose have been carefully moulded, each nostril having been stuffed with resin, which, in addition to being part of the antiseptic toilet of the face, helped to preserve the form of the nose. The lips are slightly parted and the mouth appears as a transverse fusiform slit....The opening was filled with a dark brown resinous paste, some of which was removed in 1886, exposing parts of two teeth on the right side. Both these teeth have been broken recently (? When the resin was being removed). The ears had been smeared with a thick layer of resinous paste. The greater part of the body is still enclosed in a hard shield of linen impregnated with resinous material. The scale of an onion was found adhering to the resin in the neighbourhood of the left axilla. This is of some interest, for in the succeeding three dynasties onions were used freely in the process of embalming, whether as deodorants or antiseptics, or for some unknown symbolic reason must remain a matter of conjecture. The embalming wound... margins are thickly smeared with a paste of reddish resin. The legs are still encrusted, in the greater part of their extent, with a resinous mass 6 millimetres thick. A thick resinous layer fills up the concavities of the arches of the feet. The eyes have been smeared with resinous materials, so that it is not possible to say with certainty whether or not any foreign material has been introduced underneath the eyelids" (Smith 1912, p.63-65, pl.XLII-XLIV).

More recent radiographic examination was able to show an opaque area in the cranial cavity described as *"molten resin"* applied after the removal of the brain (Harris & Wente 1980, Fig.5.9b). Although the initial studies had referred to the family's characteristic beaked nose shape having been *"carefully moulded"* and *"stuffed with resin"*, the more recent radiographical examination revealed that the shape was also due to packing the nasal cavities with peppercorns to prevent it being flattened by the wrappings (Paris 1985 in Dunand & Lichtenberg 1994, p.76). Again, with reference to the family's characteristic red hair, the initial study could not say whether the discolouration of the hair, finger and toe-

nails was “*due to the staining by resinous embalming materials [or] to henna*” (Smith 1912, p.61), although more recent in-depth analysis was able to confirm that henna had indeed been used on Ramses’ receding hair in an attempt to recreate the flaming red hair of his youth. (Paris 1985, p.256, 390). Analysis of the plant materials used in Ramses’ mummification also discovered the use of chamomile (*Anthemis tinctoria* L.), “sprinkled into the abdominal cavity... probably as an insecticide” (Paris 1985, in Manniche 1989, p.75).

The long-lived Ramses was succeeded by his 12th son Merneptah whose startlingly white mummy is that of an elderly man (Harris & Weeks 1973, p.20). Examination of his mouth revealed a “*resinous paste filling the space between the lips*” (Smith 1912, p.69, pl.XLV-XLIX) and:

“the whole body was covered in parts by a thin layer of very fine linen impregnated with a bright yellow resin-like material....which proved to be a balsam. The arms, the chest wall, parts of the leg and feet were enclosed in this balsam-impregnated carapace of fine linen. After the brain had been removed the embalmers packed the cranial cavity with small pieces of fine linen and some balsam; the nostrils were then plugged with a resinous paste and the same material was spread over the mouth and ears. A semilunar patch of black paint was then applied in the situation of the eyebrows. Beyond this a thin layer of red paste had been applied to the face. In places this has now peeled off leaving white patches. The embalming wound is almost vertical....The wound was smeared with resinous paste and a plate applied to its surface, but only part of the impression of the plate is now evident. The body had been packed with a white cheesy material, such as I found in many mummies of the priests of Amen (of the XXIst dynasty). [We] considered the material (in the case of the latter mummies) to consist of the decomposition-products of a mixture of butter and soda. Midway between the root of the penis and the anus a transverse scar is visible. It represents the place from where the scrotal sac was cut away, but as it is now thickly smeared with balsam it is not possible to say whether it was removed in life or after death. It was certainly done before the process of embalming was completed because the wound is coated with balsam. The skin of the body is thickly encrusted with salt, which [was] examined and found to be sodium chloride” (Smith 1912, p.66-68),

a fact taken by some as evidence that Menepthah was the pharaoh of the Exodus! Later radiographs confirmed the removal of the nasal septa (Harris & Wente 1980, p.168).

In the case of King Siptah, whose mummy reveals a shortening of the left leg as a result of cerebral palsy:

“Each foot was partially wrapped in a large quantity of soft muslin of exceedingly fine texture, and the surface of this covering was smeared with a thick layer of resinous paste. Each arm was wrapped in a large mass of bandages of exquisitely fine linen smeared on its surface with a layer of resinous paste. Lying on the front of the chest there was a mass of torn bandages with strips passing over the shoulders. The whole mass was thickly plastered with resinous paste, in which the impression of part of the usual pectoral ornament could be plainly seen. In front of the right elbow was a cake of resin (adherent to the bandages) in which there was a vertical groove 0m.015 mil. in diameter lined with gold foil. It was evidently the impression of a gilded staff originally placed in the left hand of the mummy. The face was entirely hidden by a thick mass of resin firmly adherent to the skin and to the cloth covering it. Some of this I removed to expose the features of the face. The abdomen was packed with lichen, and the embalming-incision... was sewn up with narrow strips of linen. In the mummy of Siptah several innovations in the technique of embalming make their appearance. The cheeks are filled out with linen packing and the body cavity filled with dried lichen. The embalming wound was sewn up.

Although this method of dealing with it is not new, since I have seen it in the XVIIIth dynasty mummy of Thuaa [Tuya], it now became the custom and apparently remained so until after the death of Ramses IV" (Smith 1912, p.71-73, pl.LX-LXIII).

Chemical analysis at the time claimed to find traces of myrrh in samples of resinous material taken from the king's face (Lucas 1908, p.142-143), with later examination noting that *"the discoloration of the skin is similar to that in Meneptah's mummy"* (Harris & Weeks 1973, p.45). Radiographs also confirmed the removal of the nasal septa (Harris & Wente 1980, p.168).

Again in the case of Seti II, the:

"Successive layers of bandage were smeared with resinous paste, and in some places quite a thick layer of material was found; it exhibited on the surface the imprints of the skin patterns of the fingers of the man who had moulded it into shape. Embedded in the resin I found around each leg a piece of string (on which a series of blue glaze "eyes" of the usual pattern, were threaded) wound spirally around the leg from ankle to knee. The body had been packed with pieces of linen soaked in a solution of resin, which set into a stone-like mass filling the whole cavity. The features were well-preserved and were not distorted, but the face is thickly encrusted with a resinous paste. The scalp was not treated in this manner. Then we found a [linen] pad on the right hip, consisting of three ragged pieces of very fine linen, one of them smeared with resin paste. Each leg had been wrapped in large quantities of very fine muslin bandages. When the leg was completely covered, a layer of resinous paste 0m.002 mill. thick was spread over the whole surface: another series of bandages was then applied and another layer of paste and so on until eight layers of alternate muslin and resinous paste (four of each were put on). The arms were treated in a similar manner and every finger and toe wrapped separately... By means of these openings they [robbers] obtained a number of small charms, but at the same time they left some of these objects, which I found firmly embedded in the resinous capsule which enclosed the two legs. The features of the face have been well preserved without much distortion; but the face is thickly encrusted with resinous material as far up as a line crossing the frontal eminences and running obliquely downward on each side to the upper margin of the external ear. Unlike the case of Siptah's mummy where the features were completely hidden and disguised by the paste covering it, the contour of Seti II's face is quite evident, and its life-like appearance is enhanced by the fact that the resin has cracked along the lines of the palpebral clefts. In the intervals where the resin has peeled off the lips, chin and cheeks no trace of hair is visible except on the right side of the mandible. The anus is plastered over with resinous material" (Smith 1912, p.74-75, 77-80, pl.LXIV-LXVI).

The mummy of an adult female perhaps to be identified as the pharaoh Tawosret was discovered in an excellent state of preservation.

"The parcel on the right foot contained a mass of epidermis mixed with large quantities of natron: that on the left portions of viscera with similar preservative material.....ordinary linen bandages, not treated with resin, were employed [to fill the body cavity]" (Smith 1912, p.82-83, pl.LXVII-LXVIII).

Recent analysis of the gilded coffins of the XIXth dynasty priestess Henutmehyt has revealed considerable information regarding her burial, and although her mummy has never been found, the thick black 'resinous' material adhering to the bottom of her inner coffin retains the back of her skull, scalp, hair and linen wrappings together with a large amount of grain (personal communication, J. Fletcher). A cache of embalming materials found near the XIXth dynasty Theban tomb of Roy contained linen bags of natron (Harris & Wente 1980, Fig.1.9), although recent analysis of similar bags has proved they contain nothing

more than common salt (Taylor 1999, p.25-27). In their examination of the mummy of the XXth dynasty pharaoh Ramses III, the investigators noted that:

“As the resin-impregnated carapace investing this mummy is quite complete, excepting the head portion, which was removed in 1886, it was deemed undesirable to interfere with it. Hence we have no direct information concerning the treatment of the body of Ramses III”

although they did add that they found artificial eyes (Smith 1912, p.86-87, pl.L-LII). Later studies state that *“an external examination indicates packing of the orbits”* together with oral packing and the removal of the nasal septa (Harris & Wente 1980, p.168,170; Harris & Weeks 1973, p.46).

In the case of the mummy of Ramses IV:

*“In front of each collapsed eye a small onion had been pushed under the eyelids to simulate the real eyes. Through the nostrils the brain was removed and the cranial cavity packed with a reddish resin in a state of powder. The nose was then packed with a resinous paste (which is now set in a mass of stony hardness), and the surface of this paste in each nostril was covered with the scale of an onion. The mouth is filled with a black resinous pate, which is also spread over the lips in a band about 13 millimetres broad. Part of this mass was loose in the space between the lips and I removed it so as to expose seven of the upper teeth. The abdomen is packed with short stalks of dried lichen, probably *Parmelia furfuracea* Ab. Precisely similar material was found in the mummy of Siptah. A plug of resinous paste was placed in the anus”* (Smith 1912, p.88-90, pl.LIII-LIV).

Chemical analysis at the time claimed to have identified myrrh in samples of the ‘*black resinous material*’ taken from the king’s cranium and mouth (Lucas 1908, p.142-143).

Whilst studying the mummy of Ramses V, the investigators noted that:

“The abdomen contained sawdust with some unrecognisable viscera lying loose (without wrappings) in it. This fact is of interest, when it is recalled that during the time of the succeeding, or perhaps even in the latter part of the XXth dynasty, it became the custom to replace the viscera in the body cavity, usually in a sawdust packing. The face was painted a red, earthy colour, like that of the mummies of many priests of the XXIst dynasty. Both nostrils were plugged with discs of wax. Linen was packed under the eyelids to form artificial eyes. The lips are placed in exact apposition [sic] and the oral cleft filled with wax. Immediately below Poupart’s ligament in the right groin there is a large irregularly triangular deep ulcer with thickened edges. It measures 0m.022 mill. by 0m. 018 mill. It is smeared with a black resinous paste, which prevents a minute examination of the characters of the ulcer” (Smith 1912, p.90-91, pl.LV-LVI).

Later radiographs confirmed the removal of the nasal septa (Harris & Wente 1980, p.168; Harris & Weeks 1973, p.20).

During the New Kingdom (1550-1069 BC) ancient Egyptian mummification reached its peak, contrary to the commonly held misconception that this occurred later during the XXIst dynasty.

To describe in general terms the ‘classic’ form of New Kingdom mummification, the body was first taken soon after death to ‘the place of purification’ (‘ibu’), a temporary tented area

within sacred temple precincts. Here it would be washed in natron salt solution then transferred to 'the house of beauty' ('per nefer'), a similar tented area in which the transformation effected by mummification would take place. In the most costly form of the process, the body would be laid out on a sloping stone embalming table, whose drainage channels would allow the body fluids and blood to drain away for collection. After washing the body with a further natron solution, a narrow metal probe would be pushed up through the nose to break the ethmoid bone and gain access to the brain, and contrary to the generally held belief that the embalmers then painstakingly scooped out the brain piecemeal, practical studies have shown that the most likely technique would involve the probe's gradual rotation which would reduce the brain to liquid after less than 30 minutes (Filce Leek, 1969). Alternatively the brain could be removed through the back of the head, after which the skull was rinsed out and filled with varying quantities of 'resins'.

The left side of the abdomen was opened using an obsidian blade and the lungs, stomach, liver and intestines removed. The heart and kidneys were left in place (Smith & Dawson 1924, Fig.38), since the heart was vital for a successful journey to the afterlife and the kidneys simply seem to have been too deeply embedded to reach. The organs removed were treated with natron and so-called 'resins' in the same way as the body (see below), before being wrapped in linen and placed in four separate 'canopic' jars. After cleaning the thoracic and abdominal cavities with palm wine and 'spices', temporary packages of natron were placed inside the hollow body shell which was then desiccated beneath piles of dry natron, rather than in natron solution as had been the case in the Old Kingdom (see above). After a period of 40 days, the natron was removed from the inside and outside of the body, which was again washed with either a natron solution or the ritually powerful Nile water.

Packing materials such as 'resin'-impregnated linen, sawdust, mud and lichen were placed in the abdominal and cranial cavities, with a variety of perfumes applied to mask the smell of decomposition (Fletcher 1998, p.55). The incision was then sewn up (Smith & Dawson 1924, Fig.36; Andrews 1984, Fig.13) and/or sealed with so-called 'resin' or beeswax and/or the addition of a gold plaque placed across it. The finger and toenails were tied onto their respective digits (Smith & Dawson 1924, p.88) or were held in place using gold finger and toe stalls (Carter 1927, p.129, 137, 151). As yet unidentified 'oils' were then applied to the body in order to keep it supple and 'resins' applied to prevent re-hydration in the relative humidity of the tombs' microclimate. The head of the deceased was often shaved and wigs

employed for reasons of ritual purity (Fletcher 2000, p.495-501), with facial cosmetics often applied to the eyes, lips and cheeks.

The body was then wrapped in varying amounts of linen layers, either specially prepared strips or recycled household linen (even in the case of certain pharaohs, Smith 1912, p.74-75). To the intoning of spells to protect the deceased, protective amulets were placed within the wrappings at specific points, with further ritual 'oils' and 'resins' applied to the wrappings at various stages, and acacia(?) gum (rather than animal glue) supposedly used to secure the bandaging. The mummified body was then placed in its coffin and liberally anointed with funerary libations (Carter 1927, p.87, 101) with the final burial occurring a prescribed 70 days after death, and accompanied by any materials which had come into contact with the body; as they contained something of the deceased they would therefore be necessary to make him/her whole in the next life.

Following a succession of ephemeral kings in the late XXth dynasty, the death of Ramses XI in 1070 BC marking the end of the New Kingdom and the beginning of the Third Intermediate Period. Egypt was once again divided as the kings of the XXIst dynasty moved north to Tanis and the Theban priest-kings ruled the south to create a confusing picture of divided rule and intermarriage between 1070-945 BC.

Although the mummy of Herihor, first priest-king of the XXIst dynasty, has remained undiscovered, that of his wife Queen Nodjmet, daughter of the last king Ramses XI, was intricately prepared.

"The back is not stuffed; but masses of sawdust wrapped in linen were placed upon each buttock; and a very large quantity of sawdust was packed around other parts of the body, and especially the legs and abdomen, and retained in position by means of bandages impregnated with resinous material so as to form a complete carapace, analogous to that seen in the mummy of Ramses III. No definite plate was placed over the embalming-wound; but an amorphous lump of wax, about the size of a hen's egg, was plugged into the wound. The eyes, mouth, nostrils and ears were protected by wax plates, which I removed in order to display the features. Artificial eyes, made of white and black stone, were inserted under the eyelids. This is the earliest instance of the use of stone eyes in a mummy....The nose was stuffed with resin and the mouth with sawdust. The cheeks are so tightly stuffed that the lower part of the face... has become almost circular. Although the introduction of foreign materials into the mouth had been in vogue ever since the time of Siptah, this is the first mummy in which the cheeks are really filled out: in the late XLXth and XXth dynasties only a small amount of packing material was introduced between the gums and lips; but from Notmit's [Nodjmet's] time onward the cheeks were tightly packed. This stuffing has elongated the upper lip...The body cavity is stuffed with sawdust....On the right arm there was the impression (in the resinous carapace) of a band-bracelet that had been stolen in modern times" (Smith 1912, p.96-97, pl.LXIX-LXXI; Harris & Weeks 1973, p.48).

The mummy of Ramses XI's other daughter Hentawy suffered from both tomb robbers and the embalmers' over-packing of the skin.

"The large hole in front of the thorax and abdomen was made by tomb-robbers in ancient times. It extends not only through the resin-impregnated carapace, but also through the wall of the body itself. As the greater part of the mummy is still encased in a hard carapace of resin-impregnated linen, only those parts where this has been broken through are available for examination. Both the cheeks and right foot were stuffed with that curious cheese-like mixture of fat (?butter) and soda, such as I have described as the packing material employed for stuffing Makeri's [Maatkare's] neck. This material was frequently employed during the XXIst dynasty for stuffing the mouth and neck; but this is the only mummy in which I have found it in the feet. An exceptionally large quantity of the cheese-like material was packed into the mouth; and with the deliquescence of the salts mixed with the fat, the stretched skin of the cheeks has burst open on each side, from the outer angle of the eye downwards to the chin. Thus her own skin has separated like a mask, which some writers have mistaken for an actual mask. The face has been painted with yellow ochre and gum and the lips (and possibly the cheeks also) have been painted red. The body-cavity is lightly packed with very fine, reddish, aromatic sawdust. In searching through as much of this material as could be reached through the wound ... I found four teeth, seven pyramidal seeds (? pine), two wax genii, two fragments of intestine and 44 beads made of gold (? or electrum). A large plug of resinous paste, in which was embedded a good deal of coarse sawdust and some of the gold wire spirals, was pushed into the embalming wound, forcing its edges apart... On the outer surface of this resin plug there was a plate of wax" (Smith 1912, p.101, 103-104, pl.LXXV-LXXVI; Harris & Weeks 1973, p.20).

The mummy of Hentawy's daughter the priestess Maatkare was studied in considerable detail, and it was noted that:

"Various foreign substances had been introduced under the skin on every part of the body and moulded into some semblance of the queen when alive... but tomb robbers had ripped through the carapace of linen from the forehead to the pelvis, so that the front of the body is hidden by a mass of torn linen, intermingled with sawdust, which has escaped from the body cavity through its damaged walls. A large quantity of mud was put into the mouth, stuffing out the cheeks so unduly as to lend an almost Eskimo-like aspect of the face. The face was painted with a mixture of yellow ochre and gum; an the nostrils were plugged with red resin. Powdered resin was also sprinkled over the face; and a sheet of muslin was then applied to it. The muslin has now become quite adherent, the gum in the paint acting as the adhesive material. The ends of many of the plaits had blobs of solid material (resinous paste) attached to them. The head was enclosed in a strong carapace of linen and resin, 11 millimetres thick, which was built up in the following manner. It was first wrapped in a sheet of muslin of exceeding fineness until a layer 3 millimetres thick was formed: to the surface of this a layer of resinous paste 4 millimetres thick was applied: to this was added a quantity of very fine linen until a layer 1 millimetre thick was formed, and finally another three millimetres of resinous paste was applied. Somewhat similar treatment had been applied to the limbs, after they had been packed in the manner described below. The carapaces of the legs are still intact: they are bound together below the knee, and across the ankles and feet by broad bandages, smeared with resinous paste. In the process of embalming.....after the body had been preserved by long immersion in a preservative bath, the embalmer introduced into the neck a quantity of fat (possibly butter) mixed with soda which is now a cheese-like mass, and with this distended the skin so as to give it the fulness [sic] of the living neck in place of the emaciated caricature seen in mummies not treated in this fashion. This cheesy material was introduced into the neck by the embalmer inserting his hand into the wound in the left flank and passing it right up through the body cavity. It was not possible (without damaging the mummy) to determine how the thoracic opening of the neck was treated, but in the other mummies of the XXIst dynasty I found linen plugs inserted so as to close the thoracic inlet. The body cavity was packed with sawdust: but no traces of viscera, funerary genii or any other object are now present in this stuffing material. The thorax thus packed was protected by a complicated series of coverings. In the process of unwrapping eight layers of very fine muslin were first removed, then a carapace of resinous paste (2 millimetres thick), then, eleven layers of fine but exceedingly closely woven linen, then a layer of resinous paste (1 millimetre thick), then another sheet of linen and another layer of resin (2 millimetres thick) and then were exposed those curious leather objects commonly referred to as "braces".....so thickly plastered with resinous material, which adheres firmly to their surfaces, that it is not possible to see the pictures or the inscriptions impressed upon the parchment....Resinous material was spread freely over these "braces" fixing them to the underlying coverings of the mummy, which consisted of the following: eight layers of moderately fine linen, covering a coating of resinous paste (2 millimetres thick), under which there was a sheet of finely and closely-woven linen, another sheet of similar texture stained red, then a thin layer of resinous paste and then two more sheets of fine (white) linen. From an incision on the shoulder fine straw-like sawdust had been packed under the skin of the arm in sufficient quantity to fill it out to the size of the living arm. In the case of the right arm the sawdust was pushed

(from the shoulder) even on to the back of the hand, but on the left side it stops at the wrist, where a natural plug has been formed by a mass of tendons pushed down from the forearm. Below this plug a quantity of coarse linen has been introduced under the skin on the dorsum of the left hand” (Smith 1912, p.98-101, pl.LXXII-LXXIII).

The smaller mummy buried with the queen and described as ‘her baby Princess Mutemhat” (Smith 1912, p.98, pl.LXXIV) was actually found to be a mummified baboon following radiographical examination (Harris & Weeks 1973, p.53, 174).

Two of Maatkare’s brothers ruled the country between them, Psusennes I as king at Tanis and Masaherta as High Priest in Thebes, a geographical division which is clearly reflected in the condition of their mummies. The damp conditions of the north had reduced Psusennes’ gold covered mummy to a skeleton whereas the mummy of Masaherta had been perfectly preserved in the arid conditions of the traditional Theban necropolis. The investigators note that Masaherta’s hair to be:

“short and white; but it is now thickly smeared with resinous material. The face and whole body was painted with a thick layer of red ochre, as was customary in the mummies of men in this dynasty. The body has been submitted to the packing-procedures customary at the time it was embalmed; but the cheeks, as usually happened were much too tightly stuffed so that they have an unnaturally puffed-out appearance” (Smith 1912, p.106, pl.LXXIX),

a feature noted in the aforementioned mummy of his mother Hentawy. It was also noted that in the case of the corpulent Masaherta:

“the embalmers departed from the custom of their time in choosing the site for the embalming incision: for instead of making it high up above the level of the iliac spine, they reverted to the custom that prevailed in the late XVIIIth and again in the early XXth dynasties and made it parallel to and alongside Poupart’s ligament” (Smith 1912, p.106).

Masaherta was succeeded as high priest by his brother Menkheperre, and although his mummy has not been found, his massive wig of office reveals the use of ‘resin’ as a fixative (Lucas 1930, p.192-193; Fletcher 2000, p.498). The mummy of Menkheperre’s wife Isimkheb, herself a daughter of Psusennes I, was found intact and has never been unwrapped since the wrappings were “so complete and perfect in every way” (Smith 1912, p.106, pl.LXXX; Harris & Weeks 1973, p.51). Their daughter Henettawy was buried in a small family tomb at Deir el-Bahari (DB.60), her mummy:

“stuffed with sawdust to fill it out, and glass eyes were placed in the eye sockets. Seven canopic packages had been put back inside the body after mummification” (Boston 1988, p.163).

In c.970 BC their son Pinudjem II became high priest, his mummy discovered largely intact and:

“enclosed, like those of Makeri [Maatkare] and Honttaoui [Hentawy] described above, in beautifully fine muslin in large quantities, with several layers of resinous paste interspersed amongst it. The

embalmers had now learned not to over-pack the cheeks...The face was sprinkled freely with powdered resin, much of which has "caked" and become adherent to the skin" (Smith 1912, p.107, pl.LXXXI).

The mummy of his wife Queen Neskhons:

"is a typical example of the distinctive technique of embalming of the XXIst and XXIInd dynasties; but its freedoms from the gross distortions of face and members that marked the earlier attempts at packing is perhaps a distinguishing mark of XXIInd dynasty work. The neck is stuffed with the cheese-like material in the manner described in the case of Makeri's [Maatkare's] mummy. There is a vertical incision.... On the antero-lateral aspect of each shoulder, from which a small quantity of packing material was introduced under the skin...The stuffing consists of sawdust; and the moulding of the arms has been skilfully done. The embalmer introduced his hand into the embalming wound in the left flank and forced the packing material (in this case a mixture of mud, sawdust and the cheese-like material, to which I have already referred), into each leg. The feet are stuffed; but I was unable to determine the spot where the packing material was introduced. Flowers were wrapped around the great toe of each foot and a flower on a long stalk was placed upon the upper surface of the left foot, and another encircled the left ankle. The embalming wound is in the situation characteristic of this period: it is the vertical incision passing from the margin of the ribs to within 0m. 035 millimetres of the anterior superior spine of the left ilium. It was covered by a wax plate of the usual form but without the usual eye design. Onion scales were placed upon the surface of the plate. The body cavity was packed with sawdust. The face is thickly encrusted with powdered resin, and large cakes of resinous material cover the eyes, nostrils and mouth. Underneath the resin shields artificial eyes of stone are found. The hair is thickly strewn with powdered red resin" (Smith 1912, p.108-109, pl.LXXXII-LXXXIV).

The mummies of other royal individuals include that of Queen Taweret, her wrappings ('carapace'):

"built up like those already described of Makeri [Maatkare] and Honttaoui [Hentawy] of fine linen and resinous paste. But the latter is freely mixed with sawdust in this mummy; and in places the fine muslin bandages are stuck together with masses of gum. The nostrils were covered with circular discs of wax; and on the sides of the nose large buttresses of wax were placed to support and prevent distortion of the nose. There is a large plate of wax in front of right eye, but none in front of the left... The lips are widely separated and the space between them filled with a large projecting mass of wax. The cheeks are stuffed like those of the three other mummies just described" (Smith 1912, p.105, pl.LXXVII-LXXVIII).

Artificial stone eyes were also employed to recreate the incredibly life-like appearance of the mummy of Lady Nesitanebasheru of the XXIInd dynasty (Ill.8).

"The face especially has been very successfully treated and the filling out of the cheeks and the artificial eyes of stone help in conveying a good idea of how this lady must have appeared in the flesh. Only a moderate amount of packing was introduced under the skin of the limbs, which on the whole were well moulded. But the body cavity was very tightly stuffed with exceptionally fine sawdust, or rather powdered wood, which still has a strong, pungent aromatic odour. The embalming wound in the left flank... was stuffed [with] a crumpled sheet of exquisitely fine linen. On the surface of this muslin was placed a lump of reddish translucent resin (0m. 087 mill. x 0m. 033 mill. x 0m. 019 mill.). There is a distinct impression upon the skin and this piece of resin of an oblong plate (0m. 122 mill. x 0m. 09 mill.). On the outer surface of each shoulder... there is a vertical incision. Through these openings the arms were stuffed. A wound upon the front of the left shoulder... reveals sawdust and a plug of linen as the materials used. The legs were packed in the usual manner and the modelling of the limbs has been accomplished in a very successful manner. The common practice of making an incision between the great and second toes for the purpose of packing the foot was resorted to in this mummy. The surface of the mummy seems to have been painted with a mixture of yellow ochre and gum, to which I have referred in the case of other mummies of women of this period. There are brown patches of discoloration upon the face, possibly due to the resin or the deliquescence of salts used in embalming. The rest of the skin is of a light yellow colour, which is not wholly due to the yellow ochre applied to it. Both nostrils were packed with resin. The upper teeth project slightly, so that their unworn edges can be seen projecting through the resin with which the rather full lips and the narrow cleft between them have been smeared. The margins of the hair-bearing areas of the scalp are thickly smeared with

resinous materials. Immediately above the right ear there is a mass of the cheese-like packing mixture of fat and soda, to which I have referred above. The packing and modelling of the neck has been unusually successful in this mummy" (Smith 1912, p.110-111, pl.LXXXV-LXXXVIII).

The body cavity of the adult male Djedptahefankh also of the XXIInd dynasty:

"was packed with lichen (Parmelia furfuracea, Ab.). The mouth is packed with sawdust. The neck has been carefully stuffed, but is not quite symmetrical. Only a small area of the back... has been stuffed: there is no packing in the buttocks....Only a small part of the legs and very little of the arms have been stuffed. The usual incision for packing the foot was found between the great and second toes. The usual crescentic area of dark brown paste is found on the brow ridge. The hair of the head was left long and was thickly plastered with resin. It is of a reddish brown colour (possibly due to partial bleaching and staining with embalming materials). The moustache and beard had not been shaven for some days before death, and this fact and the presence of much white powder and resin gives the face a very dirty and unkempt appearance. The lobules of the ears seem to have been pierced, but they are thickly smeared with resinous material...The cranial cavity is partly occupied by powdered resin, which was introduced through the right nasal fossa" (Smith 1912, p.113-114, pl.LXXXIX-XCIII).

In addition to the above mummies discovered in the two royal caches (KV.35 & DB.320), a third cache discovered in 1891 at the Deir el-Bahari site of Bab el-Gasus contained 150 mummies of priests and priestesses of the XXIst dynasty. The 44 which remained in Cairo were studied *in situ* (Smith 1912, p.106), a notable claim being the identification of myrrh in the treatment of the head of one of the priestesses (Lucas 1908, p.142-143). The rest were donated abroad and account for the majority of Egyptian mummies in world-wide museum collections, together with those individuals buried in the immediate vicinity. These include the mummy of priest Natsefamun c.1070 BC (the 'Leeds Mummy') which was unwrapped and studied in 1828, when it was noted that it *"still retains the faint smell of cinnamon....but when exposed to heat the odour of myrrh becomes very powerfully predominant"* (Osburn 1828) (discussed below). The mummy of priest Horemkenesi c.1040 BC (the 'Bristol Mummy') was unwrapped and studied more recently (Taylor 1995), with results updated within the last 12 months (Buckley & Evershed 2000) (Ill.7).

In recent X-ray and CT scan examination of several XXIInd dynasty (946-712 BC) mummies also from Thebes, that of Penu (c.800 BC) revealed artificial eyes and *"the orbits of the eyes...filled, perhaps with wax"* (Boston 1988, p.169), whereas the mummy of Ankhpefhor (c.900 BC) retained the eyes, with *"resin poured into the orbits"* and *"filling material was identified in the mouth and at the base of the neck"* (Boston 1988, p.171). The mummy of the woman Tabes (c.900 BC) was in good condition, with the eyes present, the brain removed and packing material in the throat and abdomen, which had two evisceration incisions on the left flank and across the stomach (Boston 1988, p.170), as did that of her husband, the barber Nesptah (c.800 BC). His brain had been removed and replaced with linen packing, and a:

“great deal of molten resin had been poured into his chest and abdominal cavities and packages containing the mummified viscera are embedded in this hardened resin...A package of homogeneous material, probably sand, was placed between his legs” (Boston 1988, p.220-221).

Nesptah's mummy is also typical of the XXIInd dynasty in that its case was also heavily covered in large amounts of organic material, described as the:

“curious funerary practice of ...anointing the cartonnage or mummy wrappings and occasionally the coffin with a resinous libation that has turned black in the course of the centuries.... At first glance the application of this coating seems surprising, since it obscures the carefully painted scenes and texts of the cartonnages. Although this would not have nullified the efficacy of the paintings as magical aids to the deceased, it seems likely that the resinous substance was originally transparent and that the ancient Egyptians did not anticipate the chemical changes that caused the darkening. The anointing of mummies and coffins with a resinous substance is attested in burials from the Old Kingdom to Dynasty 19 but it is unknown in Dynasty 21 and its reintroduction toward the end of the tenth century BC was perhaps connected with the change in burial customs that occurred at that time. The coffins of Nestanebasheru [mummy discussed above] which were probably prepared in early Dynasty 22, present the earliest instance of this treatment in the Third Intermediate Period. During Dynasty 22 many cartonnages were treated in this way, including that of Tabes [mummy discussed above], which has, however, been cleaned by the Museum of Fine Arts Research Laboratory. On some examples the front is completely covered with the substance, while on others, such as Nesptah's [mummy discussed above], only part of the surface is affected. The rear is usually clean and the manner in which the liquid has run down the sides indicates that the cartonnage was anointed while in a recumbent position – probably when lying in its coffin (streaks of the black substance can be observed on the inner walls of some coffins of this period). In some cases so much liquid was used that the cartonnage became stuck to the floor of the coffin. The pouring of this libation must have formed part of the funeral ceremonies immediately before the interment, but its precise significance is unknown, as is the exact chemical composition of the liquid used. In a few cases where the cartonnage was anointed, particularly thoroughly, care was taken to leave the face mask clean. The practice was abandoned about the time that cartonnages fell out of use, the latest examples dating to around the end of the eighth century” (Boston 1988, p.220; Dawson & Gray 1968, pl.VI.d; personal communication, A. Walster).

The process of mummification underwent significant changes during the XXIst-XXIInd dynasties, and in the attempt to recreate the most life-like appearance possible, faces were generally painted and the use of false hair and wigs was even more widespread than in the New Kingdom. In order to counteract the shrinkage and ‘emaciation’ which resulted from the desiccation process, a variety of materials such as mud and/or sawdust were packed underneath the skin (Smith & Dawson 1924, Fig.31, 32), sometimes so enthusiastically the packing split open the skin (Smith 1912, p.103). Body cavities previously filled with ‘resin-impregnated linen’ were instead generally stuffed with sawdust, and the treated viscera were now replaced in the body rather than into canopic containers, the four sons of Horus once depicted on the containers instead substituted with an appropriate wax figurine (Smith & Dawson 1924, Fig.50-54).

Canopic containers were reintroduced some 400 years later during the Saite Period (XXVIth dynasty, 664-525 BC), although the viscera could alternatively be wrapped and placed on the thighs (Zaki & Iskander 1943, p.246). An embalmer's cache of late Saite date was discovered in the Giza necropolis (Boston 1988, p.229), and Egypt's kings continued to be buried in the north. Although none of their burials have been survived, those of their

officials have been discovered, including that of the Saite vizier Iufaa whose mummy was recently found intact in his Sakkara tomb (National Geographic film 1999). Covered in the kind of intricate bead netting commonly used to cover the exterior wrappings, the external appearance was now of paramount importance. Although mummification was becoming more widely available, with a corresponding increase in animal mummification, the technical skills employed in the actual preservation of the body itself were in sharp decline (Walker & Bierbrier 1997, p.12). Increasingly less attention was paid to the desiccation of the body, the brain was often left *in situ* and cosmetic treatments were rarely applied.

During the XXVII-XXXth dynasties mummification techniques continued to decline, although it is ironically during this period that the account regarded as the best description of ancient Egyptian mummification was obtained by the Greek historian Herodotus on his visit to Egypt (II.84-91, Herodotus 1954, p.161). Little attention was paid to the actual preservation of the body, and beneath their wrappings mummies from this time are generally poorly preserved, despite an increasingly liberal use of 'resin' (Smith & Dawson 1924, Fig.42).

Following the invasion of Egypt by Alexander the Great in 332 BC, his Ptolemaic successors (323-30 BC) adopted Egyptian burial practices, including mummification for humans and for animals on quite a phenomenal scale (Ill.9, Ill.10). During the examination of one Ptolemaic mummy (Cockburn & Cockburn 1980, p.73) a cotton wool ball described as soaked in juniper, cinnamon and myrrh was found amongst the wrappings, its discovery raising questions regarding the import of cotton into the west at such an early date. Yet during the Graeco-Roman period the external appearance remained of paramount importance, and as the adornment of the wrappings developed into a fine art (Smith & Dawson 1924, Fig.48; Andrews 1984, figs.23, 62, 65; Walker & Bierbrier 1997) (Ill.9), the preservation of the body beneath became little more than an afterthought, often little more than a collection of disarticulated bones and cleverly modelled sticks, mud and rubbish. It was only when the Roman Empire accepted Christianity in AD 395 that mummification finally ceased, since the body was no longer required in the pursuit of eternal life. Having developed over a span of almost four millennia, the Egyptian skills of preserving a body through mummification were lost in the space of a few years and have never been recaptured.

1.2.2 Animal mummification

The ancient Egyptians are as famous for mummifying animals as well as humans (for the various animals mummified see Table 1.1), and although the practice seems to have begun simply as a means of preserving the bodies of much-loved domestic pets, the far more familiar practice of presenting mummified animals as votive offerings did not actually become widespread until the Late Period. Contrary to general assumption, the Egyptians expended as much care when preserving the bodies of animals as they did in preserving their human counterparts, as portrayed in the unusual scene in the Theban tomb of Khabekhnet (TT.2) in which Anubis attends the mummy of an enormous fish (Nims 1965, p.186). It would appear that the often startlingly life-like results (Ill.4) were achieved using much the same methods and materials, and although the exact nature of the organic materials used in the mummification of both animals and humans is still to be established conclusively, research to date has almost entirely concentrated on human mummification.

Evidence for the longest-lived animal mummifying tradition dates from the reign of Amenhotep III c.1370 BC to accompany the worship of the sacred Apis bull, the earthly embodiment of the god Ptah. At the death of every such bull, the animal would be taken to the 'embalming house of the Apis' at Memphis amidst national mourning, and there placed on a massive version of the stone embalming tables used for human mummification (Andrews 1984, Fig.18). After the embalmers had followed the specific instructions contained in the 'Apis Embalming Ritual' texts, the animal's huge mummy complete with large-scale canopic jars would be taken for burial at the Serapeum at Sakkara, and laid to rest inside its massive granite sarcophagus (Shaw & Nicholson 1995, p.35-36).

Certain smaller animals associated with particular deities also received mummification and burial, including the sacred crocodiles of the god Sobek, cats of the goddess Bastet (Andrews 1984, Fig.82), the falcons/hawks of Horus and the ibis regarded as representative of the god Thoth (Andrews 1984, Fig.81). Cats and ibis in particular were mummified in their millions by Graeco-Roman times, with recent radiographical analysis of cat mummies revealing that they were killed to order (Armitage & Clutton-Brock 1981, p.185-196). The mummified bodies of ibis, cats, dogs (Ill.4), monkeys (Reeves & Wilkinson 1996, p.185) and baboons (Smith 1912, p.98, pl.LXXIV; Harris & Weeks 1973, p.53, 174) have also been discovered in association with the burials of their owners throughout the dynastic period, to the extent of being provided with their own small coffins of wood and stone (Corteggiani 1987, No.58, p.99-100).

TABLE 1.1. Animals mummified by the ancient Egyptians

<u>ANIMAL</u>	<u>ASSOCIATED DEITIES</u>
Antelope/gazelle	Satet, Anuket
Baboon (<i>Cynocephalus</i> ape)	Thoth, Hapy, Khonsu
Bull	Ra
-Apis	Ptah, Osiris
-Buchis	Montu
-Mnevis	Ra
Cat	Bastet, Ra
Cow	Hathor, Bat
Crocodile	Sobek
Dog	
Duck	
Falcon	Ra, Horus, Montu, Sokar, Qebsenuf
Fish	
-Tilapia	Ra, Hathor
-Carp	Osiris, Hatmehyt
-Oxyrynchus	Osiris
Goose	Geb, Amun
Ibis	Thoth
Ichneumon (mongoose)	Ra, Mafdet
Jackal	Anubis, Wepwawet, Duamutef
Lion	Aker, Sekhmet, Mihos
Lizard	Atum
Ram	Amun, Khnum, Heryshef
Snake	Wadjet, Meretseger, Apophis, Renenutet
Scarab beetle	Khepri, Ra
Shrew	

1.3 EPIGRAPHIC EVIDENCE FOR MUMMIFICATION: PICTORIAL AND TEXTUAL SOURCES

1.3.1 Primary pictorial sources

Since the art of the ancient Egyptians was simply an extension of their pictorial literary script hieroglyphs, it is unsurprising that they were as reticent in depicting the mummification process as they were in describing it (see 1.3.2.1 below). The images they created were also intended to magically reanimate and exert influence upon the living, and so it is perhaps understandable that they preferred not to show what was a very bloody and, in some ways, highly destructive process. All the representations concerning mummification never actually show the process taking place, and none predate the Ramesside period of the New Kingdom (1210-1100 BC). The vast majority originate from the Theban necropolis, and whilst an unusual representation of resurrection is to be found in ceiling paintings in the tomb of Ramses IX in the Valley of the Kings in which the dead arise and throw off their linen wrappings (Boston 1988, Fig.19, p.34), almost every other scene involves a single mummy receiving attention as it lays on a funerary bier.

The most detailed images are to be found in two Ramesside tombs in which the mummy is laid flat whilst being attended to by two shaven-headed men. In the tomb scenes of Thoy, the two appear to be using brushes to apply the contents of small pots to the surface of the mummy, with a larger bowl on the floor below the body and two further bowls standing on what may be a brazier or table, as a third man appears to be reading (incantations?) from a roll of papyrus. The similar scenes in the tomb of Amenemope show two men using a brush and small pot to apply something to the surface of the mummy, as in the next scene they wind linen strips around it before applying the painted details to the mummy mask once *in situ*. Otherwise every other scene involves Anubis, the god of embalming, or a priest in an Anubis mask. The Theban tombs of Paser (TT.106) and Sennedjem (TT.1), coffin scenes of Sennedjem's son Khonsu (Saleh & Sourouzian No.216) and the funerary papyri scenes of the priestess Muthetepi (Andrews 1984, Fig.80) for example all show their respective mummies laid flat on a bier, the only attention they receive being touched (or 'resuscitated') by the Anubis figure (Boston 1988, Fig.15, p.29; Nims 1965, p.182).

A more detailed version in the Theban tomb of Nakhtamun (TT.335) shows his mummy being attended by Anubis wielding an adze, as the goddess Nephthys pours out liquid from an ankh-shaped vase at the deceased's head and her sister-goddess Isis pours out liquefied

ointment or oil over the legs (el-Mahdy 1989, p.56). An unusual variation on the theme is to be found in the Theban tomb of Khabekhnet (TT.2), in which Anubis attends the mummy of an enormous fish (Nims 1965, p.186), although in each case a number of stone vessels stand below the bier and almost certainly containing the materials used in the mummification process, three more such ointment vessels also depicted beneath the mummy of Ani in his Ramesside funerary papyri scenes (Andres 1984, Fig.12).

Scenes on the Late Period coffin of Djedbastiufankh features a number of stages in the mummification process, as the blackened figure of the deceased is washed and purified with water and with natron. His body is then laid on a bier with corn growing beneath him in what must be an allusion to the use of so-called Osiris Beds, small trays filled with seed corn and placed in the tomb to germinate in the belief that they would encourage the deceased to do likewise. Funerary rituals are then undertaken on Djedbastiufankh's behalf by three priests led by Anubis who then attends his mummy, the presence of two bags of linen below the bier suggesting he is wrapping the body. The completed mummy is then shown in the same position as the four canopic jars containing his entrails stand beneath the bier. (Andrews 1984, Fig.11; Boston 1988, Fig.5, p.15).

During the Ptolemaic Period, the scene of Anubis attending a mummy is to be found amongst temple reliefs at el-Hibis (Boston p.230, note 5), although it is otherwise restricted to funerary contexts, commonly on Graeco-Roman funerary stelae (e.g. Boston, No.186, p.230), shrouds (e.g. Boston 1988, No.153, p.203) and coffins (e.g. Andrews 1984, Fig.65). One particular version of the same appears on the footboard of a coffin of Roman date, Anubis touching the mummy with one hand whilst holding a small vessel in the other, "containing funerary oils or Nile water" (BMFA.1979.37, in Boston 1988, No.158, p.209).

1.3.2. Textual sources

To fully understand the mummification process and the materials used, the mummies themselves must be studied in conjunction with the available written information, from the tantalising first-hand clues given by the Egyptians themselves in their art and literature to the later but more comprehensive accounts of classical authors.

1.3.2.1 Primary textual sources (Egyptian)

The ancient Egyptians had developed a highly sophisticated language by c.3000 BC, and kept written records of many different aspects of their culture. It may therefore be assumed

that this extended to some form of reference to the process of embalming their dead, although in fact no written details of the procedure have been found and may reflect the secretive nature of the procedure. All that has survived are brief references in the occasional tomb inscription, literary quote and funerary text.

One of the very few references to mummification for the entire Old Kingdom (2686-2182 BC) lists the exact period of time taken for the process, and is to be found around the door of the IVth dynasty Giza tomb of Queen Meresankh III. The inscription on the right door jamb reads:

"The king's daughter Meresankh, Year 1, month 1 of the third season, day 21, her soul went to rest and she proceeded to the house of mummification (wabet)",

and then on the left jamb:

"The king's wife Meresankh, Year 2, month 2 of the second season, day 18, she proceeded to her beautiful tomb".

The total of 272 days is far longer than the standard 70 days of later times (attested for the New Kingdom and Ptolemaic periods, Smith & Dawson 1924, p.53-54), and is almost certainly to be explained by the use of natron salts in solution as found in contemporary burials (see above) rather than the more rapid results of the dry natron used later (see below; Harris & Wente 1980, p.9).

Two further texts refer to mummification in the Middle Kingdom (1985-1795 BC), and in the philosophical tract 'The Admonitions of Ipuwer', the lamentations of the author included the situation in which:

"None sail north to Byblos today. What shall we do for pine trees for our mummies? Free men are buried with their produce, nobles are embalmed with their oil as far as Crete" (Lichtheim 1975, p.152; Smith & Dawson 1924, p.56).

Mummification as a mark of status is also referred to in the Middle Kingdom story of the official Sinuhe, in which the king Sesostri I writes to his exiled courtier describing the honours which await him on his return to Egypt:

"Think of the day of burial.... with ointments and wrappings. A funeral procession is made for you on the day of burial; the mummy case is of gold, its head of lapis lazuli....The offering list is read to you....You shall not die abroad! Not shall Asiatics inter you, you shall not be wrapped in the skin of a ram to serve as your coffin....Think of your corpse, come back!" (Lichtheim 1975, p.229-230).

Two New Kingdom Theban tomb scenes list a funerary commodity as "*fat for embalming the mummy*" (Smith & Dawson 1924, p.56), and an embalmer's cache of the late XXVIth dynasty contained three bowls labelled 'ta pekheret', 'the prescription', the phrase

“applying prescriptions” referring specifically to the process of mummification, with two of the bowls labelled ‘cleansing’ and ‘natron’ (‘hesmen’) (Boston 1988, p.228). There are also useful Graeco-Roman commodity lists itemising funeral expenses (see Table 1.2) including terms translated as wax, myrrh, tallow, cedar oil, good oil and linen (Smith & Dawson 1924, p.64), a similar papyrus of AD 1st century listing cedar oil, olive oil and linen (Smith & Dawson 1924, p.65).

Rather more information may be gleaned from certain religious and funerary texts which touch upon certain aspects of mummification, but these are limited and provide little on the specific materials employed in the process. In terms of the funerary texts designed to enable the deceased individual to overcome obstacles and dangers en route to the Afterlife, neither the Pyramid Texts of the Old Kingdom (2686-2182 BC) nor the Coffin Texts of the Middle Kingdom (1985-1795 BC) make any mention of either the materials employed or the physical procedures involved, and nor does their New Kingdom successor, the Book of the Dead (1550 BC used as late as c.395 AD) [which is simply the modern term given to the large collection of 200 spells and incantations the Egyptians knew as ‘The Spell for Coming Forth by Day’, together with ‘The Book of Gates’, ‘The Book of Caverns’ and ‘The Book of that which is in the Netherworld’, and later Ptolemaic (332-30 BC) variations such as ‘The Book of Breathing’]. The only allusions to mummification in these texts are an instruction to the deceased in the Book of the Dead that they should be “*pure and clean and clad in white garments...and anointed with myrrh*” (Spell No.125, Faulkner 1985, p.33-34), and elsewhere in the Book of Gates the sun god Ra tells the dead to “*loosen your mummy wrappings and take off your head masks*” (6th hour, scene 40, Boston 1988, p.33), as reflected in a prayer to the goddess Isis to “*come and remove the bindings which are upon me*” (Hayes II 1959, p.71). Otherwise there are no Egyptian texts from any part of the dynastic period (3100-332 BC) that provide any significant information regarding the embalming procedure and materials employed therein.

TABLE 1.2. Account of funeral expenses from AD 1st-2nd Century Egypt
 (after Smith & Dawson 1924, p.64)

<u>COMMODITY</u>	<u>COST IN DRACHMAE/OBOLS</u>	
Earthenware pot		2 ob.
Red paint	4 dr.	19 ob.
Wax	12 dr.	
Myrrh	4 dr.	4 ob.
Song (dirge?)		4 ob.
Tallow		8 ob.
Linen Clothes	136 dr.	16 ob.
Mask	64 dr.	
Cedar Oil	41 dr.	
Medicament for the linen cloth	4 dr.	
Good oil	4 dr.	
Turbon's wages	8 dr.	
Lamp-wicks	24 dr.	
Cost of an old tunic		24 ob.
Sweet wine		20 ob.
Barley	16 dr.	
Leaven	4 dr.	
Dog	8 dr.	
Little mask(?)	14 dr.	
2 artabae of loaves	21 dr.	
Pine cone(?)		8 ob.
Mourners	32 dr.	
Carriage by donkey	8 dr.	
Chaff(?)		12 ob.
Total:	440 dr.	16 ob.

The earliest Egyptian texts which do give any kind of detail date from the Graeco-Roman periods (332 BC- AD 395) when Egypt was no longer governed by a native monarchy. The first are those contained in the Ptolemaic Rhind Papyri, which give an account of part of the embalming procedure. Of some relevance to the materials employed in mummification, one papyrus states:

“206 hin [1 hin = 0.47 litres] of fat were boiled, as is done for a sacred animal. Thou wast rubbed with balsam by Horus, Lord of the Laboratory. Shesmu wound with his fingers the divine bandage in order to enwrap thy body with the wrappings of the gods and goddesses. Anubis as embalmer filled thy skull with resin, corn of the gods,..... cedar oil, mild ox-fat, cinnamon oil and myrrh is to all thy members....” (Smith & Dawson 1924, p.51).

The second of the Rhind papyri is similar:

“Isis went to the burial of [name of deceased]. Fat was boiled for her as is done for the mother of the sacred animal. For her balsam was rubbed in by Horus, Lord of the Laboratory. Shesmu wound with his fingers the divine bandage in order to wrap up thy body with the bandages of the gods and goddesses. Anubis as embalmer furnishes they body with ointment and bandages....” (Smith & Dawson 1924, p.51).

Another two later texts are of Roman date (c.100 AD) and refer to the ‘Mummification Ritual’, and although the initial stages of brain removal and natron-induced desiccation are missing, there are references to the kinds of unguents applied to the body as appropriate incantations are read out. The texts themselves are written in Middle Egyptian, the classic language of specifically ritual texts employed c.2000 BC to AD 400, although the hieratic handwriting has been dated to the late first century AD or later. The more complete of the two papyri (Cairo Papyrus Boulaq III) consists of 10 pages and was used in the funerary rituals of Heter, a priest of Amun-Ra and Bastet and ‘Master of Secrets’, whereas the other copy (Papyrus Louvre 5.158) belonged to Hor, a priest sharing the same titles and a contemporary of Heter (Smith & Dawson 1924, p.45-50; Troy 1993).

Despite the late date, their ‘Mummification Ritual’ texts provide invaluable information as the only written record which details the materials employed in the embalming process. The materials listed include the textiles, metal and stones together with the oils and perfumes which were regarded as the ‘fragrance of the gods’, and ‘the sweat of the gods’. The deceased is told of the divine origin of these materials and with the application to his body his senses will be restored and his body reanimated in order to allow him to enter the Netherworld where he will be challenged and tested and ultimately judged before he can pass successfully into the afterlife. It is crucial to realise that the materials were chosen both for the symbolic qualities necessary to sustain the soul in addition to their purely practical benefits needed to preserve the body.

Specific oils for example were absolutely necessary in order to 'recreate the limbs' and give mobility:

"Take the festival oil ('hekenu') for the benefit of all your limbs. May you take the perfume so that you may unite with the great sun disk, and so it may unite with you, forming your limbs. This iber-oil which comes forth from Ra comes to you in order to create your limbs, to make your heart great, so that you may stride forth to the Great Netherworld in peace. Its iber scent is your scent in the regions of the necropolis" (Troy 1993, p.64).

The anointing oils also had protective qualities:

"Take, take Osiris, take the pine oil of the West, the pine oil which comes forth from Osiris comes to you. May it save you from your enemies" (Troy 1993, p.64).

So-called 'ointments' which supposedly contained the blood and fat of enemies would allow complete freedom of movement:

"May you take the medjet oil in the Place of Anointment so that your limbs may witness in the House of Anointment. May you come and testify among the gods so that you may go on any road you wish, to any country your heart desires. May the sweat of the gods enter into you...The sweat of the gods has come forth from Punt (Somalia). The fat of your enemies enters you. How good is your heart because of the blood of those who rebel against you" (Troy 1993, p.65).

All these texts pertain to the anointment of the head, the limbs reanimated by anointing the head with oil rather than the limbs themselves. The viscera placed in their 'canopic jars' were then covered with the so-called 'oils of the children of Horus' referring to the four gods who protected each specific organ. Evisceration of the body cavity presumably occurred in conjunction with the removal of the brain, which after having been completely pulverised was allowed to drain out of the nose once the body had been turned face down. This can also be inferred by the next step in the process which required the body to be placed precisely in this position in order to anoint the upper back with oils. Regarded as the bodily fluids of the four generations of the gods of creation, the texts refer to:

"The fluids which come forth of Ra, the spittle which comes forth of Shu, the sweat which comes forth of Geb" (Troy 1993, p.65).

Oils and fats are again identified with the slaughter of the deceased's enemies:

"To you comes moringa oil that comes forth from the Eye of Horus, and honey that comes forth from the Eye of Ra. The first of your enemies comes in order that your heart may be beautified with the fat of your foes" (Troy 1993, p.66).

The wrapping of the head also involves its anointing with ihty ('breathing') oil:

"O Mistress of the West, Lady of the East, come and breathe in the head of Osiris Heter. Let him see with his eyes, let him hear with his ears, let him breathe with his nose, let him speak with his mouth, let him judge with his tongue in the Netherworld.... O Osiris, to you comes the breathing oil, may it restore life to your mouth, sight to your eyes" (Troy 1993, p.69).

Then there was a final anointment of the wrapped head with a paste of myrrh and juniper oil

at the back of the head. In the ritual wrapping of the hands, a plant known as ‘ankh-imy’ (‘life within’), resin (?) and natron are placed in the left hand:

“The ankh-imy in your hands makes you strong. Your hand is good in the heart of Osiris, with the resin from Koptos. May you take the natron which comes forth of the Valley, which is the purification which comes from Nekhbet” (Troy 1993, p.74).

Finally the text lists the various substances used in the embalming:

“To you comes incense which comes forth from Horus, myrrh which comes forth from Ra, natron which comes forth from Nekhbet, ankh-imy which comes forth from Osiris, resin which comes forth from the Great God, gum-resin which comes forth from Wennefer, justified. May they enter into your legs...” (Troy 1993, p.77).

1.3.2.2 Secondary textual sources (Classical)

Since the ancient Egyptian primary sources give so little information concerning mummification we have to rely on secondary sources in the form of the classical writers. The most important of these are Herodotus (fifth century BC) and Diodorus Siculus (first century BC), although references to embalming occur in the work of Strabo (first century BC), Pliny (first century AD) and Porphyry (Smith & Dawson 1924, p.57-71; Harris & Wente 1980, p.19-20). After a virtual silence for almost the entire three millennia during which the Egyptians had practised mummification, the actual procedure itself is first described in c.450 BC by the Greek traveller Herodotus following his visit to Egypt (II.85-90 in Herodotus, 1954, p.160-161) after he also mentions Persian funerary practices in which they generally “*cover the body with wax then bury it*”, I.142, Herodotus 1954, p.99).

In the most important and certainly most comprehensive account of the process to have survived, three different types of mummification are described, their availability dependent upon cost.

“Mummification is a distinct profession. The embalmers, when a body is brought to them, produce specimen models in wood, painted to resemble nature, and graded in quality; the best and most expensive kind is said to represent a being whose name I shrink from mentioning in this connexion [Osiris]; the next best is somewhat inferior and cheaper, while the third sort is cheapest of all. After pointing out these differences in quality, they ask which of the three is required, and the kinsmen of the dead man, having agreed on a price, go away and leave the embalmers to do their work. The most perfect process is as follows: As much as possible of the brain is extracted through the nostrils with an iron hook, and what the hook cannot reach is rinsed out with drugs; next the flank is laid open with a flint knife and the whole contents of the abdomen removed; the cavity is then thoroughly cleansed and washed out, first with palm wine and again with an infusion of pounded spices. After that it is filled with pure bruised myrrh, cassia, and every other aromatic substance [spice] with the exception of frankincense, and sewn up again, after which the body is placed in natron, covered entirely over, for seventy days - never longer. When this period, which must not be exceeded, is over, the body is washed and then wrapped from head to foot in linen cut into strips and smeared on the underside with gum, which is commonly used by the Egyptians instead of glue. In this condition the body is given back to the family who have a wooden case made, shaped like the human figure into which it is put. The case is then sealed up and stored in a sepulchral chamber, upright against the wall. When, for reasons of expense, the second quality is called for, the treatment is different: no incision is made and the

intestines are not removed, but oil of cedar is injected with a syringe into the body through the anus which is afterwards stopped up to prevent the liquid from escaping. The body is then pickled in natron for the prescribed number of days, on the last day of which the oil is drained off. The effect of it is so powerful that as it leaves the body it brings with it the stomach and intestines in a liquid state, and as the flesh, too, is dissolved by the natron, nothing of the body is left but the bones and skin. After this treatment it is returned to the family without further fuss. The third method used for embalming the bodies of the poor, is simply to clear out the intestines with a purge and keep the body seventy days in natron. It is then given back to the family to be taken away".

In the most effective and therefore most costly process described, as much of the brain as possible was removed using an iron hook to extract it through the nostrils. In replicating this process recent studies have demonstrated that if the hook is simply rotated for approximately 20-30 minutes, the brain liquefies and can then be drained out through the nose (Filce Leek 1969, p.112-116). The cranium was then rinsed out with what are simply described as 'drugs'. At the next stage, an incision was made on the left side of the abdomen using "a flint knife" and the internal organs removed. It should however be noted that the heart and kidneys were left *in situ*, the heart regarded as the seat of wisdom and learning which would be needed in the Weighing of the Heart ceremony in the afterlife, and the kidneys too difficult to remove through the incision. After death the internal organs decompose rapidly and therefore their removal was crucial if preservation were to be achieved. The lungs, stomach, liver and intestines were removed and the cavity washed with palm-wine and spices, the alcohol in particular acting as a strong anti-bacterial agent. The hollow cavity was then filled with "*bruised myrrh, cassia and every other spice except frankincense*", such perfumed materials serving to mask and therefore deny the odour of decomposition. The incision was sewn up and the body placed 'in' natron and covered over for seventy days. The translated phrase 'in natron' was originally assumed to be natron in solution, as was the case in the Old Kingdom (discussed above), although various studies (Lucas 1932; Garner 1979; Brier & Wade 1995) have demonstrated that natron is most efficient as a desiccant when used in its dry form heaped over the body, as would certainly have been the case in Herodotus' time. Furthermore, the time taken for the body to dry out beneath natron is approximately 35-40 days, rather than the whole 70 day period stated which is the time taken for the entire process including evisceration and wrapping. This time-scale is actually mentioned in the Bible:

"And Joseph commanded his servants the physicians to embalm his father: and the physicians embalmed Israel. And forty days were fulfilled for him: for so are fulfilled the days of those which are embalmed: and the Egyptians mourned for him threescore and ten days" (Genesis 50:2-3).

Once fully dried out the body was washed and wrapped in linen strips secured in place with an adhesive 'gum'. Once placed in its coffin it was then returned to the family for burial. Herodotus' second method of mummification did not involve evisceration through an

incision, the internal organs instead dissolved by injecting 'oil of cedar' through the anus and/or vagina. After blocking these orifices to keep the oil in place, the body was then dried with natron, after which the oil was drained away along with as much of the putrefied entrails as it had 'dissolved'. The third method is even less comprehensive, with only a purge of the intestines undertaken before the body is placed beneath natron, in both cases the body then simply washed, wrapped and returned to the family for burial.

Although Herodotus' account of mummification is the most detailed to have survived, and is therefore historically invaluable, it should nevertheless be treated with caution. It was written at a time when standards of mummification were in serious decline and is at best a second-hand account, obtained by a Greek through interpreted conversations with Egyptian priests, and it is questionable how much information they would be willing to give anyone, let alone a foreigner, regarding such a deeply secretive profession. Nor is very much information given about the specific resins, spices etc. employed, and although myrrh and cassia are mentioned, the terms 'drugs' and 'every spice except frankincense' are not particularly illuminating. Palm wine is mentioned, as is 'cedar oil' - the origin and meaning of which is still widely disputed (Baumann 1950, p.84-104; Lucas 1931, p.13-21; Mendlesohn 1944, p.1795-1804) – together with 'gum', which is generally, though not conclusively, identified as acacia.

The most extensive account of Egyptian mummification second only to that of Herodotus is that of Diodorus Siculus, a Greek historian writing over four centuries later c.40 BC. He states that:

"when a person amongst them dies, all his relatives and friends, putting mud upon their heads, go about the town lamenting, until the time of burying the body. In the meantime they abstain from bathing and from wine and all kind of delicacies, neither do they wear fine apparel. They have three manners of burial: one very costly, one medium and one modest. Upon the first a talent of silver is spent, upon the second twenty minae but in the third there is very little cost. Those who attend to the bodies have learned from their forefathers. These, carrying to the household of the deceased illustrations of the cost of burial of each kind, ask them in which manner they desire the body to be treated. When all is agreed upon, and the corpse is handed over, the relatives deliver the body to those who are appointed to deal with it in the accustomed manner. First, he who is called the scribe, lying the body down, marks on the left flank where it is to be cut. Then he who is called the cutter takes an Ethiopian stone, and cuts the flesh as the law prescribes, and forthwith escapes running, those who are present pursuing and throwing stones and cursing, as though turning the defilement [of his act] on to his head. For whosoever inflicts violence upon, or wounds, or in any way injures a body of his own kind, they hold worthy of hatred. The embalmers, on the other hand, they esteem worthy of every honour and respect, associating with the priests and being admitted to the temples without hindrance as holy men. When they have assembled for the treatment of the body which has been cut, one of them inserts his hand through the wound in the corpse into the breast and takes out everything excepting the kidneys and the heart. Another man cleanses each of the entrails, sweetening them with palm wine and with incense. Finally, having washed the whole body, they first diligently treat it with cedar oil and other things for over thirty days, and then with myrrh and cinnamon and [spices], which not only have the power to preserve it for a long time, but also impart a fragrant smell. Having treated it, they

restore it to the relatives with every member of the body preserved so perfectly that even the eyelashes and eyebrows remain, the whole appearance of the body being unchangeable, and the cast of the features recognisable. Therefore, many of the Egyptians, keeping the bodies of their ancestors in fine chambers, can behold at a glance those who died before they themselves were born. Thus, while they contemplate the size and proportions of their bodies, and even the very lineaments of their faces, they present an example of a kind of inverted necromancy and seem to live in the same age with those upon whom they look” (Smith & Dawson 1924, p.62-63).

In describing the means of cutting through flesh, Diodorus’ reference to the “Ethiopian stone” describes the black colour of obsidian, which recent experiments have conclusively shown cuts easily through human flesh (as does the ‘flint’ mentioned by Herodotus), whereas the metals available at the time were far less effective and soon become blunt (Brier & Wade 1995). Diodorus follows Herodotus in his reference to the use of palm-wine, mixed with various (unnamed) spices and used for cleaning the viscera. He comments that after the body has been washed, it was then anointed with ‘cedar oil’ and ‘other things’ and “*then with myrrh and cinnamon*” and other unspecified materials in order to preserve and perfume the body. Although not part of the account, Diodorus also mentions Dead Sea bitumen and its transportation to Egypt to sell for use in embalming (Diodorus, XIX:6). It should be noted however, that he makes no mention of bitumen in his account of the mummification process, in the same way Herodotus mentions bitumen a number of times in his writings, but again never in the context of mummification. However bitumen is named in connection with embalming by Strabo, the Greek historian and geographer writing only twenty years later c.20 BC describing the Dead Sea and the bitumen it contains being used for this purpose by the Egyptians (Strabo, 16:11,45). The Roman author Pliny the Elder visiting Egypt AD c.50 recounts the use of a number of commodities in connection with mummification, and although he often refers to bitumen, he makes no mention of its use in embalming. Both the Roman writers Plutarch and Porphyry make reference to the fact that the internal organs are preserved separately, but neither mention any materials used in conjunction with the process (Smith & Dawson 1924, p.66-67).

1.4 PROBLEMS OF TRANSLATED TEXTUAL TERMS

Although the aforementioned primary Egyptian and secondary classical sources provide important information regarding the mummification process, they offer few precise means of identifying the organic embalming materials employed. The translation of the Egyptian terms (in hieroglyphs, hieratic, demotic and Coptic) together with those translated from the Greek and Latin texts are often based on little more than individual scholars’ preconceptions. The various oils used for anointing various parts of the body during the mummification process were first listed on jar labels as early as c.3000 BC, the most widely

used being the so-called 'Seven Sacred Oils' named as setiheb, hekenu, sefeti, nechenem, tewat, best ash and best tjehehu, "*although we do not know their modern names*" (Boston 1988, p.81; Fletcher 1998, p.14).

To give only brief examples of the many problems of translation which beset the subject, it is a subject of vigorous debate if the 'cedar oil' mentioned in Herodotus' account of mummification (II.90, Herodotus 1954, p161) does in fact refer to cedar or to juniper oil produced from the fruit (Baumann 1960, p.84-104; Lucas 1931, p.13-21; Mendelsohn 1944, p.1795-1804; Gale et al. 2000, p.349), a problem of identification created by the fact that the Greek word 'kedros' and the Latin 'cedrus' both mean cedar and juniper (Meiggs 1982, p.410). There are also problems with translations of the Egyptian word 'antiw', the material used as an oil to anoint the limbs and in the final anointing of the head. Some believe it to be frankincense (e.g. Hepper 1992(b), p.136; Smith & Dawson 1924, p.47, 49) whilst most scholars translate antiw as myrrh (e.g. Troy 1993, p.69, 77), with the resin known as 'senetcher' (literally 'fragrance of the gods') to be identified as frankincense (Serpico 2000, p.438). It is also possible that antiw may have had a wider meaning of gum resins in general. Similarly the identification of certain coniferous resins is of particular interest to Egyptologists and has long been disputed since the ancient word 'ash' has been variously translated as both cedar and Cilician fir (Lucas 1989, p.319-320, 439; Hepper 1990, p.20, 44-45, 47).

There are several Egyptian words for natron, i.e. 'hesmen', 'bed', 'besen' and 'netchery', the latter term linguistically related to the word for 'god' ('netcher'), and therefore known as 'the divine salt' (and also the origin of Na, the chemical symbol for sodium; Harris & Wente 1980, p.8). In terms of linguistics, it has long been argued that the Persian word 'mummiya' meaning bitumen is the origin of the modern word mummy (Lucas 1989, p.271), although it is very much less well known that the older Egyptian (Coptic) word 'mum' means wax, another of the important materials used in the mummification process (Granville 1825, p.269-316). Given that the actual ancient Egyptian word for 'the dead' i.e. the mummy was 'meni' (Gardiner 1957, A.54, p.447), it is clear that linguistic similarities existed within Egypt itself for the origin of the word 'mummy' and links with the use of bitumen are by no means exclusive.

1.5 ORGANIC MATERIALS REPUTEDLY USED IN ANCIENT EGYPTIAN MUMMIFICATION

A summary of the major organic commodities said to have been used in ancient Egyptian mummification is shown together with their ancient geographical origins in Table 1.3 and Figure 1.2. Sufficient evidence remains to establish a general identification of the nature and origin of the commodities which may have been employed in the process of mummification, although it should be noted that it can prove problematic to pinpoint exactly the ancient geographical origin of the commodities in question since many have shifted considerably as a result of overexploitation over time, and ancient texts describing their locations can also be misleading and inaccurate. Yet if firm identification can be established as a result of chemical analysis, the resulting information can provide hitherto unavailable data regarding ancient trade routes and cultural links, together with specific details of ritual procedures and technological expertise. Previous scientific research into the identification of the materials used in mummification has to a great extent concentrated on the inorganic rather than the organic materials employed, and one of the aims of this research is to redress that balance. In his summary of ancient Egyptian materials used in the mummification process first published in 1926, Lucas listed alum, beeswax, bitumen, cassia and cinnamon, cedar, henna, honey, juniper, lichen, ointments, onions, palm wine, resins, sawdust, spices, wood pitch and wood tar, although it is natron which received the most detailed attention (Lucas 1989, p.263-267, 303-326, 493-494).

1.5.1 Natron

Although not within the remit of this study the use of natron is mentioned briefly here due to its central and crucial role in the mummification process. The natron salt used to desiccate the body was obtained from the naturally occurring sources at the Wadi Natrun and el-Kab. Studies of the natron used in mummification (Lucas 1989, p.263-267, 493-494; Sandison 1963, p.259-267; Garner 1979, p.19-24; Taylor 1999, p.25-27) have shown it to consist of a natural mixture of sodium carbonate, sodium bicarbonate, sodium chloride and sodium sulphate in varying proportions ($\text{NaHCO}_3/\text{Na}_2\text{CO}_3/\text{Na}_2\text{SO}_4/\text{NaCl}$), although the important salts in this mixture are the carbonate and bicarbonate. Whilst the sodium chloride and sodium sulphate also act as desiccants and indeed do appear to have been used in the place of natron in certain cases (e.g. Taylor 1999 p.25-27), it is the alkaline salts sodium carbonate and sodium bicarbonate which are the key ingredients. In addition to their desiccant properties, they also serve to 'degrease' the body by saponification of the fats,

TABLE 1.3. Geographical origin of the major organic commodities reputedly used in ancient Egyptian mummification

MATERIAL	GEOGRAPHICAL ORIGIN OF SUBSTANCE
Coniferous (Diterpenoid) Resins	
Aleppo Pine (<i>Pinus halepensis</i>)	Palestine, Syria
Cilician Fir (<i>Abies cilicia</i>)	Lebanon, Syria, Turkey
Lebanese Cedar (<i>Cedrus libani</i>)	Lebanon, Syria, Turkey
Oriental Spruce (<i>Picea orientalis</i>)	Turkey
Stone (Umbrella) Pine (<i>Pinus pinea</i>)	Lebanon, Syria, Turkey, Greece
Sandarac (<i>Tetraclinis articulata</i>) (formerly <i>Callitris quadrivalis</i>)	Algeria, Morocco
Juniper Oil (<i>Juniperus phoenicea</i> , <i>Juniperus oxycedrus</i>)	Sinai, Lebanon, Jordan, Mediterranean, South-east Europe, Iraq
Pistacia (non-coniferous/Triterpenoid) Resins	
Chios turpentine (<i>Pistacia terebinthus</i> , <i>Pistacia atlantica</i>)	Lebanon, Jordan, Palestine, Syria, Eastern Mediterranean
Mastic (<i>Pistasia lentiscus</i>)	Palestine, Mediterranean
Gum Resins	
Frankincense (<i>Boswellia sacra</i> , <i>Boswellia frereana</i> , <i>Boswellia papyrifera</i> , <i>Commiphora pedunculata</i>)	Somalia, Ethiopia, Oman
Myrrh (<i>Commiphora</i> spp. (formerly <i>Balsomodendron</i>))	Somalia, Yemen
Galbanum (<i>Peucedanum galbaniflora</i>)	Iran
Plant Gums	
Acacia gum (<i>Acacia nilotica</i>)	Egypt
Carob (<i>Ceratonia siliqua</i>)	Egypt, Mediterranean
Gum Tragacanth (<i>Astragalus</i> spp.)	Palestine, Syria, Turkey, Iraq, Iran
Plant Oils	
Almond (<i>Prunus dulcis</i>)	Palestine, Mediterranean, South-west Asia
Balanos (<i>Balanites aegyptiaca</i>)	Egypt, Palestine, Sudan, East Africa
Castor (<i>Ricinus communis</i>)	Egypt, Sudan
Colocynth (<i>Citrullus colocynthus</i>)	Egypt, Sinai
Lettuce (<i>Lactuca sativa</i> and <i>Lactuca serriola</i>)	Egypt
Linseed (<i>Linum usitatissimum</i>)	Egypt
Moringa (<i>Moringa peregrina</i>)	Egypt, Jordan, Sudan
Olive (<i>Olea europaea</i>)	Egypt, Mediterranean, Palestine

Table 1.3 contd.	
Poppy (<i>Papaver somniferum</i>)	Egypt
Radish (<i>Raphanus sativus</i>)	Egypt
Safflower (<i>Carthamus tinctorius</i>)	Egypt, Syria, western Asia, tropical Africa
Sesame (<i>Sesamum indicum</i>)	Egypt, Mediterranean
Tiger nut (<i>Cyperus esculentus</i>)	Egypt
Animal Fats*	
Antelope/gazelle (<i>Gazella dorcas</i> , <i>Gazella soemmerringii</i>)	Egypt, Nubia, Sudan
Cattle/oxen (<i>Bos taurus</i>)	Egypt, Nubia
Goat (<i>Capra hiscus</i>)	Egypt
Goose (<i>Anser</i> spp., <i>Branta</i> spp.)	Egypt
Sheep (<i>Ovis ares</i>)	Egypt
Waxes	
Beeswax	Egypt
Spices	
Cassia (<i>Cinnamomum cassia</i> , <i>Cassia lignia</i> , <i>Cassia</i> spp. - <i>Senna</i>)	China
Cinnamon (<i>Cinnamomum zeylanicum</i> , <i>Ocotea usamarensis</i>)	East Africa, India
Dyes	
Henna (<i>Lawsonia inermis</i>)	Egypt
Safflower (<i>Carthamus tinctorius</i>)	Egypt, Syria, western Asia, tropical Africa
Others	
Animal glue/gelatin	Egypt
Bitumen	Egypt, Palestine, Syria
Honey	Egypt
Lotus Flower (<i>Nymphaea coerulea</i>)	Egypt
Palm (Date) (<i>Phoenix dactylifera</i>)	Egypt
Sawdust (cellulose/lignin)	Egypt, Near East, Greece, etc.
Storax (<i>Liquidamber orientalis</i>)	Turkey, Rhodes, Cos

* although the animal fats listed are those most commonly used after the necessary refining ('sweetening') process, a range of rather more unexpected sources are listed in medical texts, including lion, crocodile, hippopotamus, snake, mouse etc. (Fletcher 1998, p.17; Serpico & White 2000, p.408).



KEY

AP Aleppo Pine	Li Linseed
CF Cilician Fir	Mo Moringa
LC Lebanese Cedar	Ol Olive
OS Oriental Spruce	Po Poppy
SP Stone (Umbrella) Pine	Ra Radish
Sn Sandarac	Sa Safflower
J Juniper	Ss Sesame
CT Chios Turpentine	Tn Tigernut
Ma Mastic	AF Animal Fats
F Frankincense	Bx Beeswax
My Myrrh	Cs Cassia
Gb Galbanum	Ci Cinnamon
Ac Acacia Gum	Hn Henna
Cb Carob	AG Animal Glue
GT Gum Tragacanth	Bt Bitumen
Al Almond	Ho Honey
Ba Balanos	LF Lotus flower
Ca Castor	Pa Palm (Date)
Co Colocynth	Sw Sawdust
Le Lettuce	St Storax

Fig. 1.2 Map of Near East showing the geographical origin of the major organic commodities reputedly used in ancient Egyptian mummification

followed by dissolution of the resulting lipids, to produce an emaciated mummy which is essentially skin and bone. Yet if protected from moisture the body can survive almost indefinitely in this 'degreased' form, and may be the reason the Egyptians regarded natron in both its dry and liquid form as a means of 'purifying' the body.

The amount of scholarly attention given to natron is unsurprising, given its crucial role in desiccation, yet it must be stated that the organic preservatives were of equal importance. They prevented both bacterial action and rehydration of the body, which in turn would promote autolysis and bacterial degradation (Cockburn et al. 1998, p.77). It is therefore all the more perplexing that these vital materials have not received a proportionate amount of attention, a situation the present study seeks to begin to rectify.

1.5.2 Coniferous resins

Coniferous resins were not indigenous to Egypt and had to be imported from neighbouring regions. The coniferous trees which produced the resin 'exudates' are mainly located in Sinai, Palestine, Lebanon, Jordan, Syria, Turkey, Greece and Iraq. A number of species grew in these regions, each producing greater or lesser quantities of resin when 'wounded' by a sharp implement inserted through the bark to allow the liquid resin to be collected. The likely sources include the Aleppo pine (*Pinus halepensis*), the Cilician fir (*Abies cilicia*), Lebanese cedar (*Cedrus libani*), Oriental spruce (*Picea orientalis*) and the Stone (or Umbrella) pine (*Pinus pinea*). Their resins serve to protect the trees by retaining moisture and acting as insecticides and bactericides. It is therefore unsurprising that these same resins have been connected with mummification, since such properties would be beneficial for the preservation of the body; if the body was first desiccated, the resins would act as a barrier and prevent moisture from entering the body. A resin identified as "probably from a coniferous tree and possibly cedar resin" (Lucas 1908, p.142-143) was discovered in association with an Old Kingdom burial at Giza (G.1206 C), and more recent examination of resinous material from the 1st dynasty burial of King Djer at Abydos proved to be imported cedar or pine (Serpico & White 1996, p.136).

The varying quantities of resin produced by each species have tended to be regarded as a way of eliminating the Egyptians' likely use of certain species of conifer, with the small amount of resin some produce (e.g. cedar) regarded as 'uneconomical' (Lucas 1989, 319-322). Yet to use such a relatively modern concept is somewhat misguided and ignores the fact that symbolism played a central role in concepts of life and death, certain flora and

fauna carrying a significance which could not be measured solely in terms of economic cost. The fact that a particular species of conifer produced only a small quantity of resin cannot be used as a valid argument against its use in what was considered a crucial process.

The Egyptians' use of Sandarac (*Tetraclinis articulata*) resin is generally considered unlikely "given its North African origin" (Lucas 1989, p.321; Serpico & White 2000, p.431), although it must be said Algeria and Morocco are closer to the Nile Valley than either Persia (the sole source of galbanum, see 1.5.4) or India and China (the sources of cinnamon and cassia, see 1.5.9). The use of juniper resin as opposed to cedar must also be considered here, given the controversy surrounding their use in mummification. Although it is not a resin-producing conifer, juniper does contain the diterpenoids (see Chapter 2) present in the resin-producing trees, and its berries have been found interspersed amongst the linen wrappings of mummified bodies, including Tutankhamen (Hepper 1990, p.21, 60). It is also uncertain if the 'cedar oil' mentioned in Herodotus' account of mummification (II.90, Herodotus 1954, p.161) is actually juniper oil produced from the fruit (Baumann 1960, p.84-104; Lucas 1931, p.13-21; Mendelsohn 1944, p.1795-1804; Gale et al. 2000, p.349), a problem compounded by the fact that identical Greek and Latin terms meant both cedar and juniper (Meiggs 1982, p.410, and above). The oil produced from pressed juniper berries does contain the terpenoids beneficial for body preservation, quite apart from any symbolic significance that juniper may have had. The identification of the particular coniferous resins employed has long been disputed, given confusion surrounding translations of the ancient terms used (see above) and the difficulties distinguishing between species, since the compounds in the fresh resins which differentiate between them have not been preserved in the archaeological material. Yet in favourable circumstances certain species such as the cedar can be identified (see Chapter 2).

1.5.3 Pistacia resins

The Pistacia resins have also been associated with the mummification process. These triterpenoid resins come from the regions of the Mediterranean and Near East (see Table 1.3, Fig.1.2) and originate from two main sources. One is so-called 'mastic' (also confusingly associated with bitumen) resin from the small evergreen shrub *Pistacia lentiscus* (Hepper 1990, p.26), the resin-producing variety now restricted to the island of Chios, off the coast of Turkey (Hepper 1990, p.26). The other resin is 'Chios turpentine' extracted from *Pistacia atlantica* (often mistakenly referred to as *P. terebinthus*), a large deciduous tree which ironically is more widespread than mastic and not restricted to the

island of Chios. The terpenoids present in the resins of these species would have similarly desirable properties of the conifer resins when used in the mummification process.

1.5.4 Gum resins

The gum resins reputedly used by the Egyptians were essentially frankincense, myrrh and galbanum, which in addition to their resinous (triterpenoid) portion, also contain a 'gum' (i.e. sugar) component. Both myrrh (*Commiphora* spp) and frankincense (*Boswellia* spp) originate from the region around Somalia (the Horn of Africa) and southern Arabia (Hepper 1969, p.66-72), with galbanum cultivated in the area of modern Iran (Persia). The ancient Egyptians make frequent mention of the 'antiw' and 'senetcher' translated as both frankincense and myrrh (see above) (Serpico 2000, p.438) which they obtained from 'Punt', an area generally believed to be in the region of Somalia. Relief scenes at the Deir el-Bahari funerary temple of Hatshepsut (1473-1458 BC) portray an Egyptian trading expedition arriving in Punt, with both the resin and sapling trees prepared for shipment back to Egypt (Dixon 1969, p.55-65).

Both antiw and senetcher are listed as ingredients in the perfumed oils used to 'anoint the limbs of the gods' and royalty were similarly anointed during sacred rituals of protection. The aforementioned Hatshepsut is described with "*the best of myrrh(?) on all her limbs, her fragrance is divine dew, her odour is mingled with that of Punt*" (Fletcher 1998, p.54). The belief that these gum resins were the very essence of the gods imbued them with incredible power which could be harnessed for the benefit of the deceased in the afterlife, hence their value as part of the mummification process. Myrrh was 'identified' (see Chapter 2) following analysis of samples taken from several New Kingdom royal mummies, from the cheek and left arm of Amenhotep III, the face of Siptah, the cranium of Ramses IV and the face and scalp of an unnamed XXIst dynasty priestess (Lucas 1908, p.142-143). In practical terms, their powerful scent would mask the unpleasant odours of decomposition, in addition to which the phenolics and terpenoids would act as antibacterials. The ancient Egyptians used 'antiw' preparations to freshen the breath and treat tooth pain (Fletcher 1998, p.23), with frankincense currently used in the Arabian peninsula as an antibacterial to treat digestive problems and chewed because 'it is good for the teeth and gums' (Abercrombie 1985, p.486).

Galbanum resin was similarly used in perfume production and has been identified as part of a beeswax mixture employed as part of the mummification process during the Ptolemaic Period (332-30 BC) (Benson et al. 1979, p.119-131).

1.5.5 Plant gums

These sugar-based gums have been connected with the securing of the mummy wrappings, the most commonly cited being acacia gum obtained from small trees or shrubs of the genus *Acacia* native to Egypt. Said to have been used in the place of animal glue (Baumann 1960, p.84-104), a report on the wrappings of an ancient Egyptian mummy would question this assumption with the discovery of a protein indicative of gelatin (Benson et al. 1979, p.124). Carob (*Ceratonia siliqua*) could also have been employed as an adhesive, and was available both locally and from the Mediterranean region. Notably, the carob pod was used as a determinative in the ancient Egyptian word 'nedjem' meaning 'sweet' (Gardiner 1957, M.29, p.483). Although not indigenous to Egypt, Gum tragacanth (*Astragalus*) may have been imported from Palestine, Syria and Turkey, with further cultivations in Iraq and Iran.

1.5.6 Plant oils

The plant oils available to the ancient Egyptians included almond, balanos, castor, colocynth, lettuce, linseed, moringa, olive, poppy, radish, safflower, sesame and tiger nut. All were derived from native sources with the exception of the almond, cultivated in Palestine, the Mediterranean and South-west Asia. These oils formed the base for more costly and exotic ingredients both in the domestic perfume industry and in the preparation of complex ritualistic unguents, as documented in medical papyri and related literary texts (Fletcher 1998, p.25-33). The various oils were frequently used to protect both the gods' statues and royalty, whose skin is described as "*gilded and shining as do the stars before the whole land*" (Fletcher 1998, p.54). Given their glistening qualities they were believed to render the wearer immune to the powers of darkness, either in life or after death where their use within a funerary context is obvious, so much so that the Egyptians honoured the deity Merhet solely as 'goddess of the embalming oils'.

1.5.7 Animal fats

The Egyptians employed animal fats for a wide range of domestic, ritualistic and funerary purposes, the fats' inherent odour having first been neutralised by the refining process ('sweetening') in which wine and/or wine-soaked plant matter (cyperus or coriander) were added. The most readily available sources of animal fat included domesticated cattle/oxen,

goats, sheep and geese, although medical texts also list the use of antelopes/gazelles, cats, mice and hedgehogs and the rather more exotic lions, crocodiles and hippopotami. These animals were originally indigenous to Egypt, although the number of lions were depleted by wide-scale hunting during the New Kingdom. It seems almost certain that when applied to the body as an unguent, these animal fats had symbolic significance (Lucas 1989, p.312), particularly when hedgehog fat was advocated as a cure for baldness and snake fat was believed to increase suppleness of the body (Fletcher 1998, p.17). Their characteristic attributes were believed to be present in their fat, which was therefore harnessed for beneficial purposes, and given the aforementioned importance of symbolism in mummification, it is quite possible that the same fats would be thought to imbue the deceased with the particular animal's qualities (e.g. strength, speed, bravery, etc.).

1.5.8 Waxes

Beeswax was the only wax considered to have been available to the ancient Egyptians, with apiculture practised at a very early date (Shaw & Nicholson 1995, p.51; Serpico & White 2000, p.409-411). Both wax and honey (see below) were utilised widely, and given the significance of the bee, the symbol of Lower (northern) Egypt, the sign of kingship and emblematic of the sun god Ra, the potential for the use of beeswax in mummification is obvious. In practical terms, its hydrophobic nature would also prove most useful as a barrier after the thorough desiccation of the body.

1.5.9 Spices

The two spices mentioned by both Herodotus and Diodorus in connection with mummification are cassia and cinnamon. Cassia (*Cinnamomum cassia*, *Cassia* spp.) originates from China and was certainly available during the Roman Period (Lucas 1989, p.308), and cinnamon (*Cinnamomum zeylanicum*), which is similar to cassia and similarly derives from the ground bark of certain laurels, was imported from India during the Roman period. *Ocotea usamarensis*, a similar cinnamon based product, must also be mentioned given its East-African source, and it may have been utilised during dynastic times when it is generally supposed that cassia and cinnamon would not have been available. As in the case of the highly fragrant gum resins, the phenolics present in these spices would have served to limit microbial putrefaction as well as masking the resulting unpleasant odours, their use in perfuming the body cavities being an eminently sensible one.

1.5.10 Vegetable dyes

Vegetable dyes have often been connected with mummification, their use associated with colouring both the mummified body and its wrappings. The orange/red dye henna obtained from the native Egyptian shrub *Lawsonia inermis* has recently been found as early as c.3400 BC (Fletcher 1998, p.21; Fletcher 2000, p.500), and it is still widely used in Egypt and across the Muslim world to colour the hair, hands and feet for both aesthetic and protective reasons. The native Safflower (*Carthamus tinctorius*) has also been associated with the mummified body with its use in the mummy wrappings (Hepper 1990, p.32), most notably identified in mummy wrappings from the Middle Kingdom (1985-1650 BC)(Serpico & White 2000, p.394). The dyes would have provided desirable aesthetic qualities for the deceased, with the golden yellow colour in particular echoing that of the sun god Ra with whom the deceased wished to merge.

1.5.11 Bitumen

Natural bitumens are petroleum products resulting from the loss of the more volatile components of crude oil. The remaining heavier fraction is natural 'bitumen', which can occur as seepages from source rocks, or solid blocks such as those of the Dead Sea, where the conditions have removed more of the volatiles and undergone more extensive oxidation and polymerisation. The use of bitumen in mummification, if applied instead of the 'resin', would provide a hydrophobic barrier that would prevent the absorption of moisture present in the tomb environment, assuming that the body was thoroughly desiccated prior to application. Since the word 'mummy' may derive from the Persian word for bitumen ('mummiya'), its use must clearly be considered. Although many have suggested that the name originated from the mistaken belief that the black colour of many of the mummies was due to bitumen, the extent of its use has long been disputed (Lucas 1914, p.241-245; Reutter 1912, p.45-67; Spielmann 1932, p.177-180; Griffiths 1937, p.703-709; Rullkotter & Nissenbaum 1988, p.618-621; Connan & Dessort 1991, p.1445-1452; Bahn 1992, p.109) and presently there remains much confusion surrounding its use.

The sources of bitumen potentially available to the ancient Egyptians include those of Egypt itself at Helwan (Aufrere 1991, p.640; Forbes 1964, p.25-26; Abraham 1960, p.214) and Gebel Zeit (Mons Petrolius), and although there is currently no direct evidence for their use, these deposits were close to Egypt's earliest settlements c.4000 BC. Therefore the general assumption that the Egyptians must have imported all their bitumen might seem somewhat presumptuous. Otherwise there were large deposits of bitumen across the Near

East, including those of Palestine, Syria and Iraq, in particular the Dead Sea area which has long been a familiar source of bitumen. The local people in these areas were fiercely protective of their valuable resource, and the trade routes for bitumen were frequently contested in ancient times. Although certain reports have found evidence of bitumen in mummification materials (see Chapter 2), the most recent research shows that many of the assumptions concerning its use are erroneous and must clearly be revised (Buckley & Evershed, forthcoming).

1.5.12 Honey

Honey was available in Egypt from the earliest times (see wax above, Shaw & Nicholson 1995, p.51; Serpico & White 2000, p.409-411) and was one of the key ingredients in many of the treatments listed in ancient medical papyri (Nunn 1996, p.148). The aforementioned importance of the bee as a symbol of the sun god imbued honey with significant symbolic qualities, whilst its antibacterial properties recently exploited in modern medicine made it a highly valued commodity in mummification. Indeed, the Egyptian pharaoh Alexander the Great (332-323 BC) is said to have been embalmed in honey prior to his burial in Egypt, and there is evidence of its use in Ptolemaic mummification (Benson et al. 1979, p.119-131).

1.5.13 'Lotus' flowers

The blue lotus, the name given to the native aquatic water lily *Nymphaea coerulea*, was one of the most potent symbols of ancient Egypt (Hepper 1990, p.11, 16; Nunn 1996, p.157; Fletcher 1998, p.18). It was the heraldic emblem of Upper (southern) Egypt and as the symbol of rebirth its connections with resurrection gave it immense power in the process of mummification. Lotus flowers were often laid on the mummified body and also make up funerary wreaths and bouquets placed around the tomb (Newberry in Carter 1927, p.190-196, pl.XXXVI; Osburn 1828). Its sweet fragrance was believed to be both protective and restorative and thought sufficiently strong to revive the deceased, hence representations of individuals inhaling both the blossoms and/or the extracted perfume.

1.5.14 Date palm

The native date palm (*Phoenix dactylifera*) was used by the Egyptians for a variety of purposes (Gale et al. 2000, p.347-348), including wine production. Although it has been stated that palm wine was produced by fermentation of the tree sap rather than the dates (Murray et al. 2000, p.592-593), others argue that the fruit was also used in wine production

(Lucas 1989, p.23; Manniche 1989, p.133-134). Since the composition of the fruit is quite different from the sap of the trunk (see Chapter 2), it should be possible to differentiate between the two, although in most cases there is unlikely to be sufficient material left to determine this. 'Palm wine' is mentioned by both Herodotus (II.83, Herodotus 1954, p.160) and Diodorus (Smith & Dawson 1924, p.63) as the means by which the eviscerated body cavity was rinsed out, the alcohol content acting as a highly anti-bacterial sterilising agent.

1.5.15 Sawdust

Although sawdust was used to pack various parts of the body (see chronology of mummification above), its mention here is pertinent since the lignin component of wood, which makes up about 25% of the lignocellulose biopolymer of which wood mainly consists (Galletti & Bocchini 1995, p.815), is susceptible to degradation via hydrolytic and oxidative processes (Mills & White 1994, p.80). This yields phenolic components such as coniferyl alcohol, which are the building blocks of the lignin polymer. Yet these components and their degradation products can also derive from balsamic resins (e.g. gum benzoin (Schroeder 1968, p.57), *Styrax officinalis* (Lucas 1989, p.95) and certain plants of the *Umbelliferae* family (Serpico 2000, p.450). It is therefore necessary to take this into account when determining the materials used, since the significance of sawdust would be very different from that of an imported exotic plant species with potential implications for trade routes.

1.5.16 Storax

The balsamic resin storax is obtained from the tree *Liquidamber orientalis* native to the islands of Cos and Rhodes, and the mainland of southwest Turkey. It has long been connected with mummification (Hepper 1990, p.47-48; Gale et al 2000, p.342), its phenolic and terpenoid components (see Chapter 2) again having antibacterial properties beneficial for body preservation. *Styrax officinalis*, a small shrub or tree from the eastern Mediterranean (Serpico 2000, p.437), has also been suggested as the source of the ancient 'storax/styrax resin' (Hanbury 1857, p.465), although the modern plant produces no resin.

1.6 SUMMARY OF THE ARCHAEOLOGICAL AND HISTORICAL EVIDENCE FOR MUMMIFICATION

The information derived from careful observation of the physical remains together with surviving images and texts is extremely valuable to the understanding of mummification in ancient Egypt. Yet of the embalming materials themselves (the organic 'resins', 'spices', etc. in particular) surprisingly little is known as many questions remain unanswered. Since the embalming process evolved and regressed with the rise and fall of ephemeral symbolic influences at certain periods (e.g. Amenhotep III, Akhenaten), it can be highly misleading to extrapolate backwards or forwards in time or to simply assume that the process of mummification continued to improve through time. The picture is yet further complicated by the fact that there were also various forms of the process practised at any given time, depending on the individual's status, geographical location or even personal preferences. Hence, it is of paramount importance that mummies dating from all periods of ancient Egyptian history and from as wide a geographical area as possible are examined equally. Only by undertaking such an extensive study can we hope to have a meaningful understanding of the 4000-year old history of mummification and the wide variety of materials employed.

Given problems of translation which are often based on individual scholars' preconceptions, the extant texts can offer very few certainties regarding the nature of the organic embalming materials employed. With few Egyptian equivalents, the Greek translations of the ancient Egyptian terms can be highly misleading, particularly given that the secondary texts of Greek authors were obtained at a time when ancient Egyptian mummification was long past its technical best. It must also be stressed that the pictorial evidence is of little value in attempting to identify what are amorphous organic mixtures with no visual characteristics present to identify them. Furthermore, it is crucial to understand that ritualistic considerations were no less important than practical ones and during the mummification process each particular commodity was believed to imbue the deceased with its own magical qualities. Yet if the chemistry of these commodities and the effect they would have on the body can begin to be understood, it may become possible to ascertain why the Egyptians chose to use them.

Astounding as it may seem, the understanding of the organic materials used in mummification has hardly changed since the days of pioneer Egyptologist Gardiner

Wilkinson, whose description of mummification published in 1854 is little different to those currently produced. The terms 'resin' and 'bitumen' continue to be used widely, indiscriminately and often erroneously, with little regard for the particular resins, oils, perfumes, etc. which may actually have been employed. Yet chemical analysis is ideally suited to the characterisation and identification of otherwise amorphous residues of unknown origin, and the chemical analysis of samples taken systematically from mummies can actually provide many of the answers to questions which have existed since mummification itself ceased to be practised.

CHAPTER 2

Chemistry of the organic embalming agents

CHAPTER 2: CHEMISTRY OF THE ORGANIC EMBALMING AGENTS

The ancient Egyptians mummified their dead as a response to the natural processes of human decomposition. They believed that the survival of the body was vital if they were to pass successfully into the afterlife, a belief which was to result in a remarkably sophisticated procedure of preserving their dead. One of the most important stages in the process was the application of specific organic materials which protected against oxidation, rehydration and putrefaction, and masked the unpleasant odours of decomposition. In order to appreciate both why and how the specific resins, oils and spices were utilised in mummification, it is first necessary to understand the actual processes of decomposition which the ancient Egyptians found so deeply disturbing and did all they could to prevent.

2.1 THE PHYSICAL AND BIOCHEMICAL PROCESSES OF DECOMPOSITION OF THE BODY

The natural processes involved in the decomposition of human remains are highly efficient, and within a relatively short period of time can reduce the body to little more than bone. Such processes begin immediately after death, as within an hour or so the skin begins to take on a darkened pink or purple colour on the underside of the body as blood begins to accumulate (hypostasis) through gravity. At the same time areas supporting the weight of the body become paler as the blood is squeezed out of the capillaries by the pressure exerted upon them (Barber 1988, p.104, Mims 1998, p.120). Whilst the body is still warm, muscles undergo contraction (rigor mortis), beginning with the small muscles of the jaw, fingers, eyes and mouth at between one to four hours after death, then the larger muscles of the torso and limbs after four to six hours. After 36 to 48 hours the muscles then relax as the body becomes cold (Mims 1998, p.120). Exact times for these processes vary however, and depend on both the physical condition of the individual and the environment around them. Rigor mortis is accelerated in warm conditions, as would have been the case in ancient Egypt. The process of decomposition of a body above ground is also twice as rapid as that in water and four times as fast as that underground, with corpses preserved for longer the deeper they are buried (provided the ground is not waterlogged) (Mims 1998, p.120).

As decay continues, the action of anaerobic bacterial enzymes cause the intestines to putrefy. Intestinal microbes (clostridia, coliforms etc.) begin to spread through the rest of the body, where autolysis has also begun to occur as a result of enzyme action and other chemicals released from dead tissue (Hunter et al. 1996, p.63-78). The digestive enzymes of the pancreas are particularly active and soon digest the organ itself. As putrefaction accelerates, fats, carbohydrates and proteins are broken down and the tissues begin to produce green substances and gases, causing the skin to blister and loosen and take on a blue/green colouring which spreads out from the abdominal area. The tongue often protrudes and both the mouth and nose can emit fluids from the lungs, and as the front of the body swells, hydrogen sulphide, methane and traces of mercaptans are emitted (Mims 1998, p.120).

Neutral fats in the body undergo hydrolysis by bacterial enzymes and the lipases released from the tissues. Palmitic, oleic and stearic acids are released, with the relative amount of palmitic to oleic increasing as the oleic acid is subject to hydration and oxidation processes. If the neutral fats are completely converted to fatty acids these can remain as adipocere, a mixture of largely palmitic, oleic and stearic acids along with their calcium salts (see below). In the presence of atmospheric oxygen, however, bacteria and fungi can also bring about the oxidation of body fats. Unsaturated fatty acids (largely oleic) are oxidised to aldehydes and ketones, in addition to hydroxy-, oxo- and di-fatty acids (Gulaçar et al. 1989, p.61-72; Evershed 1992, p.253-265). Archaeological studies have revealed a wide range of products which can result from lipid degradation, depending on the particular environment to which the bodies are exposed (Gulaçar et al. 1990, p.691-705; Evershed 1992, p.253-265). Usually however, there is relatively little oxygen, resulting in hydrolysis being the dominant process (rather than oxidation), and the degraded fats remaining as adipocere. Enzymes also serve to degrade the proteins present, the rate depending on temperature, moisture and microbial action. Moisture and heat promote protein breakdown, hydrolysing them to polypeptides and amino acids.

Within a few weeks, the hair, nails and teeth become detachable and after a month or so tissues have become liquefied through autolysis and microbial action. The abdominal and thoracic cavities burst open, the skin cracks and the facial features are no longer recognisable. Since moisture greatly accelerates decay, these processes can occur within hours of death in tropical areas and between four to six days in temperate regions, whereas putrefaction is dramatically inhibited in both freezing conditions and in extremely dry

environments such as those encountered in southern Egypt. Putrefaction is similarly slowed down if the body is placed in a coffin, although skin decomposes more rapidly on areas of the body which rest against the coffin, generally exposing the back of the skull, shoulder blades and tip of the vertebrae when the corpse is laid on its back or whichever areas rest against the coffin, as noted in the Egyptian pre-New Kingdom practice of laying the body on its left side (ill.3).

Within a year the body is reduced to little more than a skeleton although the bones still contain organic materials and can remain greasy for many years (Mims 1998, p.123). It can take around fifty years for them to become completely desiccated in a coffin or fairly dry soil, in which they can survive almost indefinitely in cool temperatures. Skeletal material is best preserved in neutral soil conditions, the acidity of the soil in tropical regions combining with the heat and moisture to greatly accelerate bone decomposition. Similarly in the waterlogged conditions of European peat bogs, the acidity of the soil acts with the moisture in anaerobic conditions to reduce the bones within 25-100 years, the process again accelerated if temperatures are warm (Barber 1988, p.107-108; Mims 1998, p.123; Evershed & Connolly 1988, p.143-145; Evershed 1990, p.139-153). Wet conditions can also convert the subcutaneous body fats by saponification into adipocere, a waxy, greasy material with a rancid, cheese-like odour. This can be greenish-white in colour, or brown to black if the body is in a coffin. It takes months to form and eventually becomes brittle and chalky, in which state it can persist for centuries (Barber 1988, p.108; Evershed 1992, p.253-265).

Although skin colour changes after death, from an initial white pallor with localised areas of dark pink and purple (hypostasis) and then to the green-blue or brown shades of putrefaction, the final colour of the skin depends on a range of factors. White fungal growths may appear on the skin as a result of fungal spores being present in any cosmetic preparations used during the embalming process (Mims 1998, p.123), although the “*whitish spots*” on the upper back and shoulders of Tutankhamen proved to be “*common salt with a small admixture of sodium sulphate, in all probability derived from the natron used in the embalming process*” (Harris & Wente 1980, p.17). The more overall white colour in the case of certain other mummies (Harris & Weeks 1973, p.45) are again almost certainly the result of generous natron treatment, although the white encrustations of sodium chloride covering the mummy of Merneptah (Smith 1912, p.65-68) were originally cited as proof that he was the pharaoh of the Exodus(!).

The skin of naturally mummified bodies is generally brown and leathery on account of the natural heat of the surrounding conditions, which preserved the body before the microbes had sufficient time to utilise the moisture necessary for putrefaction. Other bodies can take on a darker, blackened colour for a variety of reasons, one AD 16th century account reporting that "*the corpse was undamaged, fresh and complete – only the skin of the chest and head looked blackish, because in putting him into the coffin they had strewn him with quicklime, so that he would be consumed faster*" (Barber 1988, p.102). In the case of Egyptian mummies, the blackened colour has only occurred with their exposure to air after they were unwrapped, as noted in the case of Yuya and Tuya (Harris & Weeks 1973, p.19), Tutankhamen (Carter 1927, p.113 Pl. XXXI) and Seti I (Smith 1912, p.57; Harris & Weeks 1973, p.18). A similar process appears to have occurred in the decomposition of the liquids used on both the bodies and coffins which are said to have blackened over time (Boston 1988, p.220; Carter 1927, p.87, 101).

In addition to natural internal decomposition, the body if left untreated above ground can be consumed by a wide range of insect and animal predators, attracted by the pungent odour of decomposition. Rats, mice, dogs and flesh-eating birds can quickly dismember a corpse, spreading the remains over a wide area, the Egyptians' deification of the jackal and vulture as protective figures of the dead an attempt to subvert the creatures' otherwise destructive nature.

The most common form of insect predators are flies (*Diptera*). Also known as 'sarcophagus' or carrion flies (Mims 1998, p.121), these were again transformed by the ancient Egyptians into symbols of persistence. The flies in question are generally the bluebottle or blowfly (*Calliphora erythrocephala*), together with the more specialised flesh eaters such as greenbottles (*Lucilia*) or the coffin fly (*Conicera*). Laying their eggs around the body's orifices or skin wounds, a single fly can lay up to 2,000 eggs, the larvae (maggots) beginning to feed as soon as they hatch by secreting a powerful enzyme to dissolve the skin. Although the corpse can be completely obscured by maggots within 24 hours in tropical environments, the process is less rapid in more temperate regions (Mims 1998, p.122).

As a variety of other types of insect from beetles (*Dermestes frischii*, *Dermestes ater*) to mites (e.g. stored-product mite *glycyphagus*) begin to feed on different parts of the body and consume the soft tissue, the body can be reduced to a skeleton within a year. The

remains of such insects are often to be found within mummified remains when their life cycle was dramatically terminated by the embalming process (Smith & Dawson 1924, Fig.42; Taylor 1995, Fig.63, p.95). Alternatively they can indicate a more recent infestation as they actively consume mummified remains in museum collections (personal communication, J. Fletcher).

It was in response to such dramatic and potentially disturbing natural processes that the ancient Egyptians mummified their dead. It is also notable that when the procedure was at its most efficient (during the New Kingdom (c.1550-1069 BC), the first step involved the removal of the brain and viscera, including the intestines, the origin of putrefaction in the body (Mims 1998, p.120).

2.2 THE EFFECT OF EMBALMING AGENTS ON THE PRESERVATION OF THE BODY

Much of the previous scientific research into the identification of the materials used in mummification has focussed upon the inorganic rather than the organic materials employed. A particular focus of study has been natron, the desiccant which also removes fats from the body and thereby reduces the potential for putrefaction (Lucas 1989, p.263-267, 278-281, 493-494; Sandison 1963, p.259-267; Garner 1979, p.19-24; Taylor 1999, p.25-27). The studies have shown the key ingredients of natron to be the alkaline salts sodium carbonate and sodium bicarbonate, with the most effective and successful embalming procedures resulting from its application in dry form. Given its crucial role in desiccation, the amount of scholarly attention natron has received is unsurprising, yet the organic preservatives were of equal importance. Their application prevented bacterial action and the rehydration of the body, which in turn would promote autolysis and bacterial degradation (Cockburn et al. 1998, p.77). It is therefore all the more perplexing that these vital materials have not received a proportionate amount of attention, a situation the present study attempts to begin to rectify.

In order to assess the choice of organic materials employed, it is necessary to understand the effects such substances would have on the decomposing body. True resins such as cedar, pine, fir and *Pistacia* contain unsaturated terpenes, their anti-microbial properties (Briggs & Eglinton 1994, p.907-912) serving to eradicate the bacteria responsible for putrefaction. Gum resins, such as frankincense and myrrh, contain phenolic compounds

which are both antiseptic and anti-bacterial, their strongly aromatic qualities shared with balsamic resins also masking unpleasant odours emitted by the decomposing body.

In addition to such resins' antiseptic and/or antibacterial properties, their application on the desiccated skin and abdominal cavity interior would also prevent the rehydration which would otherwise take place in the humidity of the tombs' microclimate, resulting in its subsequent decomposition (Cockburn et al. 1998, p.77; Hunter et al. 1996, p.63-78). Oxidation of the body lipids can similarly be obviated by providing a physical barrier between the tissue and the atmospheric oxygen necessary for the oxidative processes to occur. It should be noted however that if resins were applied to a body which had not been thoroughly desiccated prior to their use, any residual moisture would then be trapped within the body, facilitating the process of bacterial putrefaction and degradation.

Yet it is crucial to understand that symbolism played as great a role in ancient Egyptian funerary practices as it did in daily life, with ritualistic considerations as important as practical ones. During the mummification process, substances were applied to a series of specific spells and incantations (see Funerary Texts, Chapter 1), with each particular commodity imbued with its own magico-religious qualities of benefit to the deceased. If the chemistry of the substances used in mummification can therefore be understood, together with the effect they would have on the body, it becomes possible to begin to understand why the Egyptians may have used them.

2.3 CHEMISTRY OF THE ORGANIC MATERIALS REPUTEDLY USED IN ANCIENT EGYPTIAN MUMMIFICATION

Table 2.1 summarises the chemistry of the organic materials reputedly used in mummification and lists the characteristic compounds present in each particular commodity together with the characteristic 'biomarkers'. These specific biological marker compounds are both resistant to degradation and are characteristic of the particular organic substance, their crucial feature being that the original carbon skeleton is preserved in a form which is sufficiently recognisable that it can be used to determine the origin of these otherwise amorphous organic residues of unknown origin. Any compounds which may be present in the commodity but are not exclusive to it are omitted.

TABLE 2.1. The major organic commodities reputedly used in ancient Egyptian mummification

MATERIAL	CHARACTERISTIC COMPONENTS	CHARACTERISTIC BIOMARKERS
Coniferous (Diterpenoid) Resins Aleppo Pine (<i>Pinus halepensis</i>) Cilician Fir (<i>Abies cilicia</i>) Lebanese Cedar (<i>Cedrus libani</i>) Oriental Spruce (<i>Picea orientalis</i>) Stone (Umbrella) Pine (<i>Pinus pinea</i>)	Diterpenoids (abietic, dehydroabietic, isopimaric, pimaric, sandaracopimaric acids) Sesquiterpenoids (substituted benzocycloheptene, substituted methanoazulenes & atlantones in cedar), longiferone Cis-abienol (characteristic of fir)	Dehydroabietic acid Dehydrodehydroabietic acid 7-Oxodehydroabietic acid 15-Hydroxy-7-oxodehydroabietic acid (Isopimaric acid*, pimaric acid*, sandaracopimaric acid*) Retene (in pitch) Substituted benzocycloheptenes Substituted methanoazulenes (e.g. cedrol) Atlantones/bisabolones Longiferone, longifolene Cis-abienol/manoyl oxide
Sandarac (<i>Tetraclinis articulata</i>) (formerly <i>Callitris quadrivalis</i>)	Communic acid (70% - as polymer) Phenolics (sandaracopimaric acid, 12-acetoxy-sandaracopimaric acid)	<i>cis</i> - and <i>trans</i> -Communic acids (Sandaracopimaric acid) 12-acetoxy-sandaracopimaric acid
Juniper Oil (<i>Juniperus phoenicea</i> and <i>Juniperus oxycedrus</i>)	Diterpenoids: pimarane, abietane, communic acids Cedrene, cedrol, thujopsene C ₁₂ & C ₁₄ fatty acids, C ₁₀ , C ₁₂ & C ₁₄ 8,ω-dihydroxycarboxylic acids C ₂₀ , C ₂₂ , C ₂₄ , C ₃₀ and C ₃₂ fatty acids Indene derivative	<i>cis</i> - and <i>trans</i> -Communic acids Dehydroabietic acid Cedrene, cedrol 8,10-Dihydroxydecanoic acid 8,12-Dihydroxydodecanoic acid 8,14-Dihydroxytetradecanoic acid C ₂₀ , C ₂₂ , C ₂₄ , C ₃₀ & C ₃₂ fatty acids
Pistacia (triterpenoid) Resins Chios turpentine (<i>Pistacia atlantica</i> [or <i>Pistacia terebinthus</i>]) Mastic (<i>Pistacia lentiscus</i>)	Triterpenoids: moronic, oleanonic, masticadienonic & isomasticadienonic acids Triterpenoids: moronic, oleanonic, masticadienonic & isomasticadienonic acids	Moronic acid, oleanonic acid, masticadienonic acid, isomasticadienonic acid Moronic acid, oleanonic acid, masticadienonic acid, isomasticadienonic acid
Gum Resins Frankincense (<i>Boswellia sacra</i> , <i>Boswellia frereana</i> , <i>Boswellia papyrifera</i> , <i>Commiphora pedunculata</i>)	α- & β-Boswellic acids α- & β-Acetyl-boswellic acids Sugars	α- and β-Boswellic acids α- and β-Acetyl-boswellic acids 11-oxo-β-boswellic acid Sugars

Table 2.1. contd. Myrrh (<i>Commiphora</i> spp. (formerly <i>Balsomodendron</i>)) Galbanum (<i>Peucedanum galbaniflora</i>, <i>Ferula gumosa</i>)	Furanosesquiterpenoids: furanoeudesma-1,3-diene (19% of essential oil – hexane extract)(unstable) furanoeudesma-1,4-dien-6-one (unstable) T-Cadinol, bisabolenes Commic acids Sugars	Disabolane/bisabolenes Sesquiterpenoids (cadalenes) Commic acids Sugars Umbelliferone (7-hydroxy coumarin)
Plant Gums Acacia gum (<i>Acacia nilotica</i>) Carob (<i>Ceratonia siliqua</i>) Gum Tragacanth (<i>Astragalus</i> spp.)	Triterpenoid glycosides Triterpenoids: dihydroxyoleanolic acid or its lactone, acacic acid lactone Sugars: esp. arabinose & galactose Sugars: mannose>galactose Sugars: arabinose, galactose, xylose, fucose	Acacic acid lactone dihydroxyoleanolic acid dihydroxydihydrooleanolic acid Arabinose, galactose Mannose, galactose Sugars
Plant Oils Almond (<i>Prunus dulcis</i>) Balanos (<i>Balanites aegyptiaca</i>) Castor (<i>Ricinus communis</i>) Colocynth (<i>Citrullus colocynthus</i>) Lettuce (<i>Lactuca sativa</i> and <i>Lactuca serriola</i>) Linseed (<i>Linum usitatissimum</i>) Moringa (<i>Moringa peregrina</i>) Olive (<i>Olea europaea</i>) Poppy (<i>Papaver somniferum</i>)	Triacylglycerols, (High C _{16:0} FA cf. C _{18:0} FA) β-Sitosterol, stigmasterol, campesterol, etc. Ricinoleic acid (12-hydroxy-9-octadecenoic acid) +4% long chain fatty acids (C ₂₀ +)	Mono-, di- and triacylglycerols Fatty acids (C _{14:0} , C _{16:0} , C _{18:1} , C _{18:0} , etc) Hydroxy- & di- fatty acids β-Sitosterol, stigmasterol, campesterol, stigmastan-3-one Ricinoleic acid (12-hydrox-9-octadecenoic acid) 9,12-Dihydroxyoctadecanoic acid +4% long chain fatty acids (C ₂₀ +)

Table 2.1. contd.		
Radish (<i>Raphanus sativus</i>)	Long-chain fatty acids - major fatty acids: erucic acid (C22:1) and arachidic acid (C20:0)	Long-chain fatty acids - major fatty acids: erucic acid (C22:1) and arachidic acid (C20:0)
Safflower (<i>Carthamus tinctorius</i>)		
Sesame (<i>Sesamum indicum</i>)		
Tiger nut (<i>Cyperus esculentus</i>)		
Animal Fats		
Antelope	Triacylglycerols, (Relatively high C18:0 FA cf. C16:0 FA). Branched chain fatty acids,	Mono-, di- and triacylglycerols
Cat		Fatty acids (C14:0, C16:0, C18:1, C18:0, etc)
Cattle/oxen		branched chain fatty acids
Crocodile	C18:1 ^Δ 9,10,11,13,14,15,16 FA's isomers	C18:1 ^Δ 9,10,11,13,14,15,16
Goat	in ruminants	FA's isomers in ruminants
Goose	Cholesterol, cholesterol esters	Cholesterol, cholesterol esters,
Hippopotamus		Cholesta-3,5-dien-7-one
Lion		7-Ketocholesterol
Sheep		
Snake		
Waxes		
Beeswax	C23-C35 alkanes (C27 dominant) C22-C36 FA's (C24 dominant) C38-C52 (C16:0 FA) wax esters C40-C54 (C16:0 FA) hydroxy wax esters	C23-C35 alkanes (C27 dominant) C22-C36 FA's (C24 dominant) C38-C52 (C16:0 FA)) wax esters C40-C54 (C16:0 FA)) hydroxy wax esters
Spices		
Cassia (<i>Cinnamomum cassia</i> , <i>Cassia lignia</i> , <i>Cassia</i> spp. - <i>Senna</i>)	<i>trans</i> -Cinnamaldehyde & cinnamyl alcohol Coumarin & δ-cadinene (trace in cinnamon)	Cinnamic acid, hydrocinnamic acid, hydroxyhydrocinnamic acids, coumarin, δ-cadinene/cadinenes
Cinnamon (<i>Cinnamomum zeylanicum</i>)	<i>trans</i> -Cinnamaldehyde & cinnamyl alcohol, Eugenol & benzyl benzoate (trace in cassia)	Cinnamic acid, hydrocinnamic acid, hydroxyhydrocinnamic acids, eugenol, benzyl benzoate
Dyes		
Henna (<i>Lawsonia inermis</i>)	Lawsone (2-hydroxy-1,4-naphthoquinone)	Lawsone (2-hydroxy-1,4-naphthoquinone)
Safflower (<i>Carthamus tinctorius</i>)	Carthamin (red) Unknown (yellow)	Carthamin 4-hydroxybenzoic acid 2,3,4,6-tetrahydroxybenzoic acid
Others		
Aloe (<i>Aloe succotrina</i>)	Aloe-emodin anthrone	Aloe-emodin anthrone
Animal glue/gelatin	Proteins	Amino acids

Table 2.1. contd.		
Bitumen	C ₁₅ + alkanes, phytane, pristane, hopanes, moretanes, steranes	C ₁₅ + alkanes, phytane, pristane Hopanes: 28-norhopane, 29-norhopane, hopane Moretanes, steranes
Honey	Sugars (glucose & fructose)	Glucose, fructose
Lotus Flower (<i>Nymphaea coerulea</i>)	Anthocyanins (anthocyanidin galloyl-galactosides)	Anthocyanidins
Palm (Date) (<i>Phoenix dactylifera</i>)	C _{12:0} fatty acid	C _{12:0} fatty acid
Sawdust (cellulose/lignin)	Syringyl and guaiacyl moieties	Syringyl and guaiacyl moieties
Storax (<i>liquidamber orientalis</i>)	Aromatic compounds: cinnamyl cinnamate, 3-phenyl-propanyl cinnamate Triterpenes: oleanonic acid, 3-epi-oleanolic acid	Cinnamic acids Oleanonic acid 3-Epi-oleanolic acid

* Susceptible to degradation but can survive in favourable environments

2.3.1 Chemical transformations

Although it can be difficult to predict the fate of the components in these complex organic materials, particularly when they may have been admixed with other substances at elevated temperatures, it is nevertheless useful to consider the chemical transformations they are likely to have undergone, particularly the diagnostic compounds. Processes which often occur in archaeological samples include oxidation, hydration, dehydration, decarboxylation, esterification, aromatisation, hydrolysis, transesterification (wax esters), hydrogenation and polymerisation. Factors which will affect the chemistry of these materials include heat (e.g. anthropogenically derived), humidity, pH, and microbial breakdown. Since the majority of mummified remains are no longer *in situ*, the likely environment must be taken into account if a true picture of the organic materials employed is to be achieved, and using a biomarker approach in which the basic carbon skeleton of the characteristic molecules remains intact in a recognisable form can in fact do this.

2.3.2 Coniferous resins

The coniferous resins derive from trees of the *Coniferae*, within which are the *Pinaceae* and *Cupressaceae* within the ancient Egyptian sphere of influence and trading network. The *Pinaceae* include pine, firs, cedars and spruces, the *Cupressaceae* include sandarac and juniper (see Table 2.1), their resins exuding when their bark is pierced. The chemical composition of the resins is dominated by terpenoids (Mills & White 1994, p.98-103; Mills & White 1977, p.12-31), with monoterpenes and diterpenoids both present in abundance.

The monoterpenes are volatile and so are likely to have been lost, although they can be trapped in the matrix of the hardened resin in some circumstances (Mills et al. 1984/85, p.15-39). They also occur in the majority of natural resins, and since they are not particularly diagnostic (Mills & White 1994, p.95) they are not considered here. The diterpenoid acids present in the fresh *Pinaceae* resins have either abietane or pimarane type skeletons (see Fig. 2.1). Both types are tricyclic, with the abietane acids containing conjugated double bonds whereas the pimarane acids lack this conjugation, a quaternary carbon separating the two double bonds present. This lack of conjugation in the pimaradiene acids makes them more stable than those of the abietadienes. These diterpenoids produce a 'soft' resin which does not undergo extensive polymerisation (see juniper and sandarac, below), and under favourable archaeological conditions these

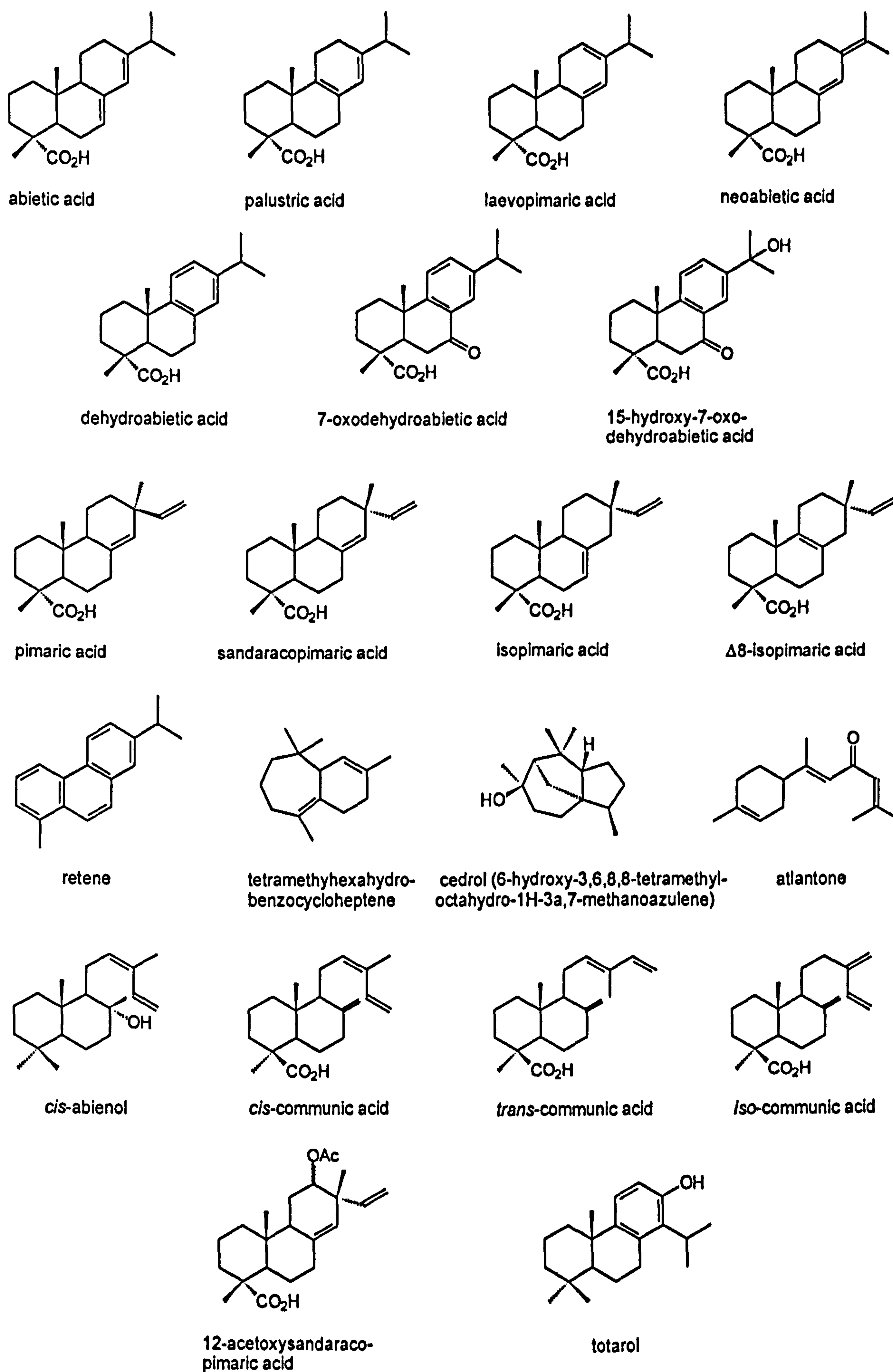


Figure 2.1 The structures of diterpenoid and sesquiterpenoid components occurring in coniferous resins. Although they do not occur in the fresh resins, 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid are often the major extractable components in aged archaeological samples.

pimarane and abietane type acids can survive relatively unchanged in coniferous resins (Mills & White 1977, p.15; Buckley & Rose, 2000 unpublished data). Yet in many archaeological samples the pimarane diterpenoids present in the fresh Pinaceae are often absent, and of the abietane compounds only dehydroabietic acid remains. Dehydroabietic acid is normally only present as a minor component in the fresh resins, but increases in abundance on ageing at the expense of the abietadiene acids as they undergo oxidative dehydrogenation to the more stable aromatic triene, dehydroabietic acid. Where oxygen is available, dehydroabietic acid can be oxidised to 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid, these diterpenoid compounds often being the dominant components remaining in highly degraded archaeological resin samples (Serpico & White 1998, p.1037-1048; Proefke et al. 1992, p.105A-111A; Weser et al 1998, p.511A-516A; Beck et al. 1999, p.281-293).

Where the resin has been subjected to strong heating, extensive aromatisation (via thermal dehydrogenation) and decarboxylation of the acids can take place, resulting in the production of compounds such as retene (see Fig. 2.1). It is the presence of aromatised diterpenoids such as retene, along with significant quantities of the thermally produced methyl esters of the diterpenoid acids, which suggests that a pitch (i.e. strongly heated resin) may have been utilised (Beck et al 1999, p.281-293; Evershed et al 1985, p.528-530; Robinson et al. 1987, p.637-644). Although species identification of resins is difficult and can often prove impossible in archaeological samples, some Pinaceae resins can be identified through chemical analysis in certain favourable environments. One such example is the Aleppo pine (*Pinus halepensis*), due to the appreciable amount of sandaracopimaric acid relative to the amount of pimaric acid and this being the more abundant in the majority of resins. Since these acids are of equal stability, the greater abundance of sandaracopimaric compared to pimaric acid (~8:1) (Mills & White 1994, p.101) can thus be used to identify aleppo pine.

Cedar (*Cedrus libani*) is also a resin in which the characteristic components, the sesquiterpenoid substituted azulenes, benzocycloheptenes, and atlantones, can be preserved, two examples of these found in cedar being the substituted methanoazulenes (e.g. cedrol) and tetramethylhexahydrobenzocycloheptene (Serpico & White 1998, p.1041; Serpico 2000, p.447) and when these survive in favourable conditions, their presence confirms a cedar resin. Cilician fir (*Abies cilicia*) is also a potential resin source which has a characteristic chemical marker, *cis*-abienol. This labdane-type alcohol (see Fig 2.1) is a

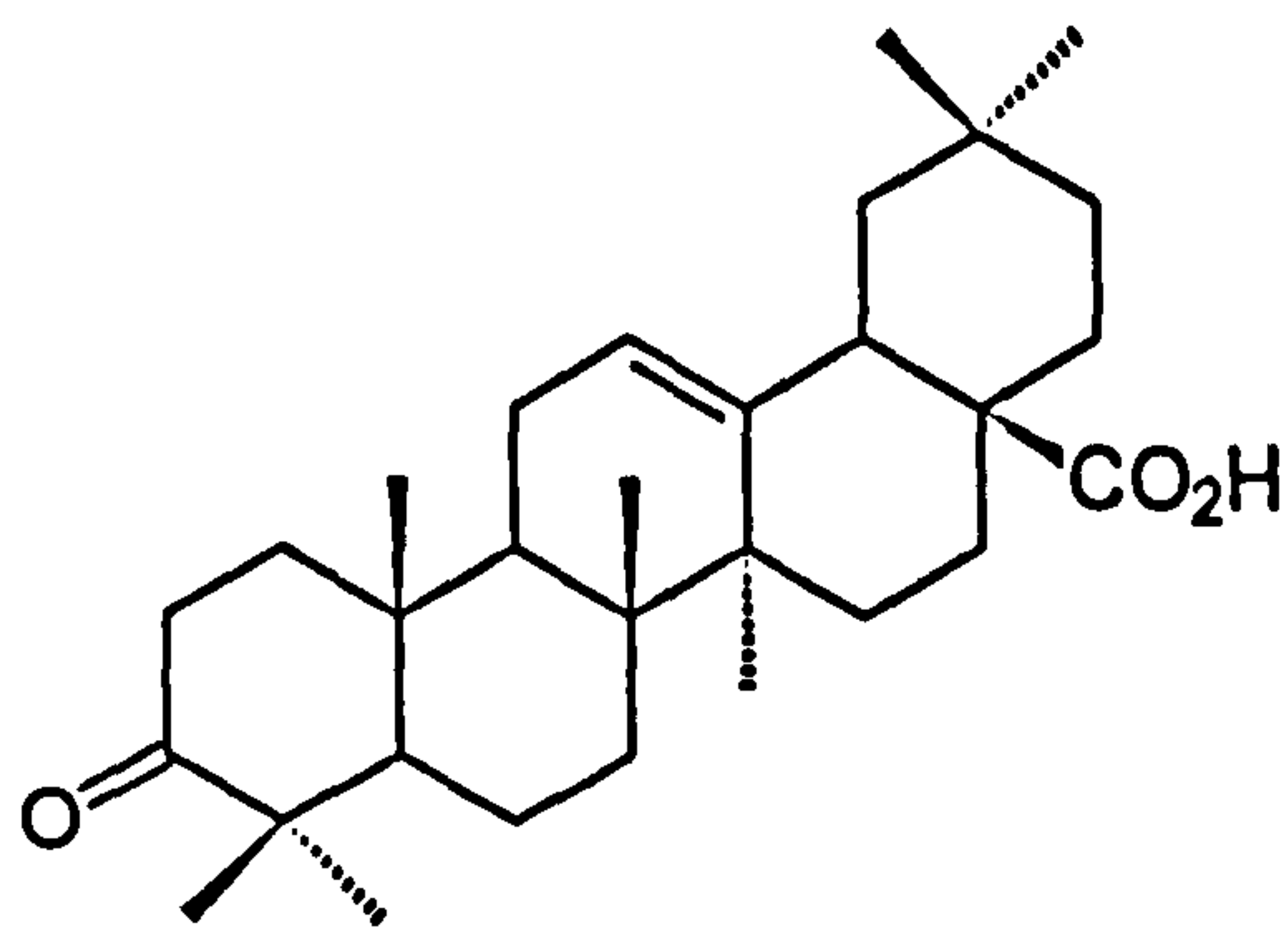
significant component of fir resin (Mills & White 1994, p.102) and its presence, or that of its degradation components, provide evidence for the use of fir (Mills & White 1977, p.17).

Of the Cupressaceae family, sandarac (from *Tetraclinis articulata*) contains the labdanoid communic acid (see Fig. 2.1) which makes up about 70% of the resin in its polymeric form (Gough 1964, p.2059-2060). The main monomeric diterpenoids present are sandaracopimaric acid, together with lesser amounts of 12-acetoxysandaracopimaric acid, and phenols including totarol (see Fig 2.1) are also present (Mills & White 1994, p.102). Juniper resin (from *J. phoenicea* & *J. oxycedrus*) again contains large quantities of polycommunic acid, in addition to pimarane, minor amounts of abietane diterpenoid acids and totarol. Juniper resin (and oil) also contains the sesquiterpenoids cedrene, cedrol and thujopsene. Due to the quantities of polycommunic acids in these two resins they are, in contrast to the Pinaceae resin, relatively hard.

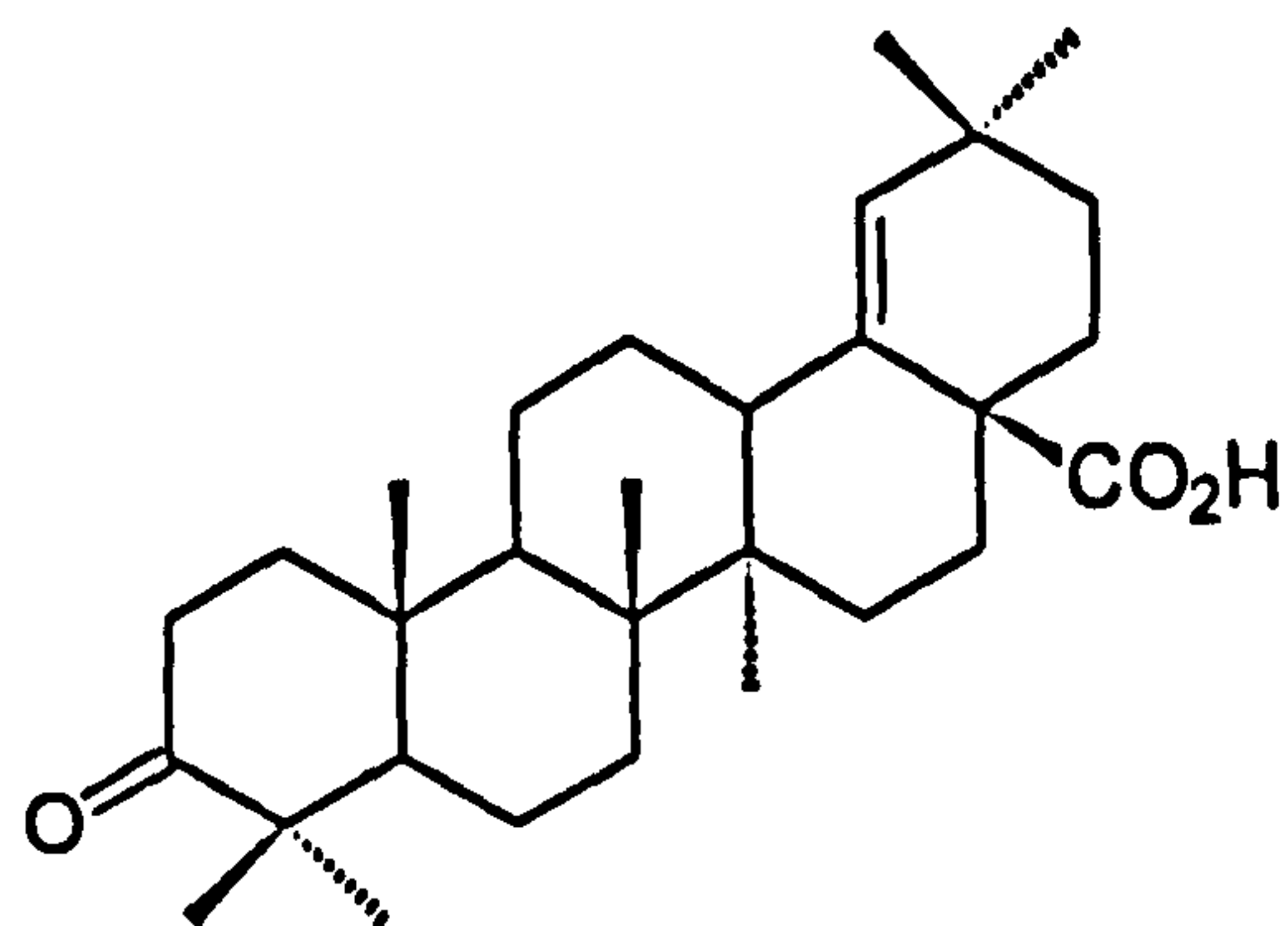
2.3.3 Pistacia resins

There are two sources of these resins, mastic (*Pistacia lentiscus*) and Chios turpentine (*Pistacia atlantica*), both of which contain triterpenoids in addition to the monoterpenoids which are ubiquitous in true resins (see Table 2.1). It is the triterpenoid acids present in these resins which are the characteristic components likely to survive in archaeological samples of the resin (Mills & White 1977, p.21; Mills & White 1989, p.37-44; Mills & White 1994, p.107-108).

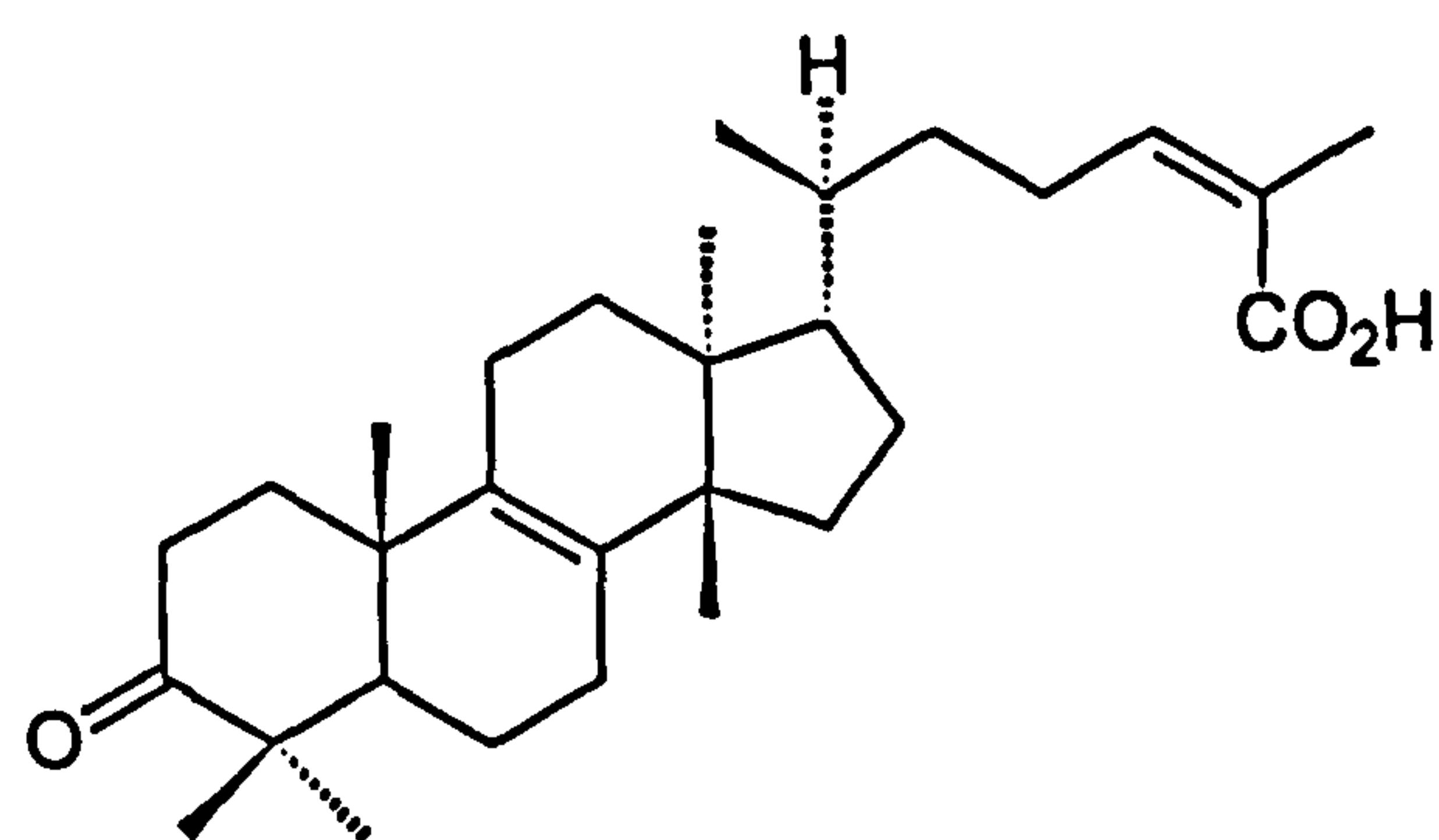
The composition of mastic has been studied in some detail, particularly the triterpenoids (Marner et al. 1991, p.3709-3712; Papageorgiou et al 1997, p.263-273), which include the neutral triterpenoids β -amyrin (which occurs widely) and tirucallol together with the triterpenoid acids oleanonic, moronic, isomasticdienonic acid and masticdienonic acid (see Fig. 2.2). The diagnostic acids moronic, isomasticdienonic and masticdienonic characteristic of Pistacia resins have been found in a number of aged and archaeological samples (Mills & White 1989, p.37-44; van der Doelen et al 1998, p.21-37; Mills & Serpico 1998 p.1037-1048; Colombini et al. 2000, p.19-29). Like other triterpenoid resins such as dammar, Pistacia resins also contain a polymeric hydrocarbon component (Mills & White 1994, p.108). Given that the composition of the two Pistacia resins is very similar (Mills & White 1989, p.37-44; Serpico 2000, p.447) it is not usually possible to



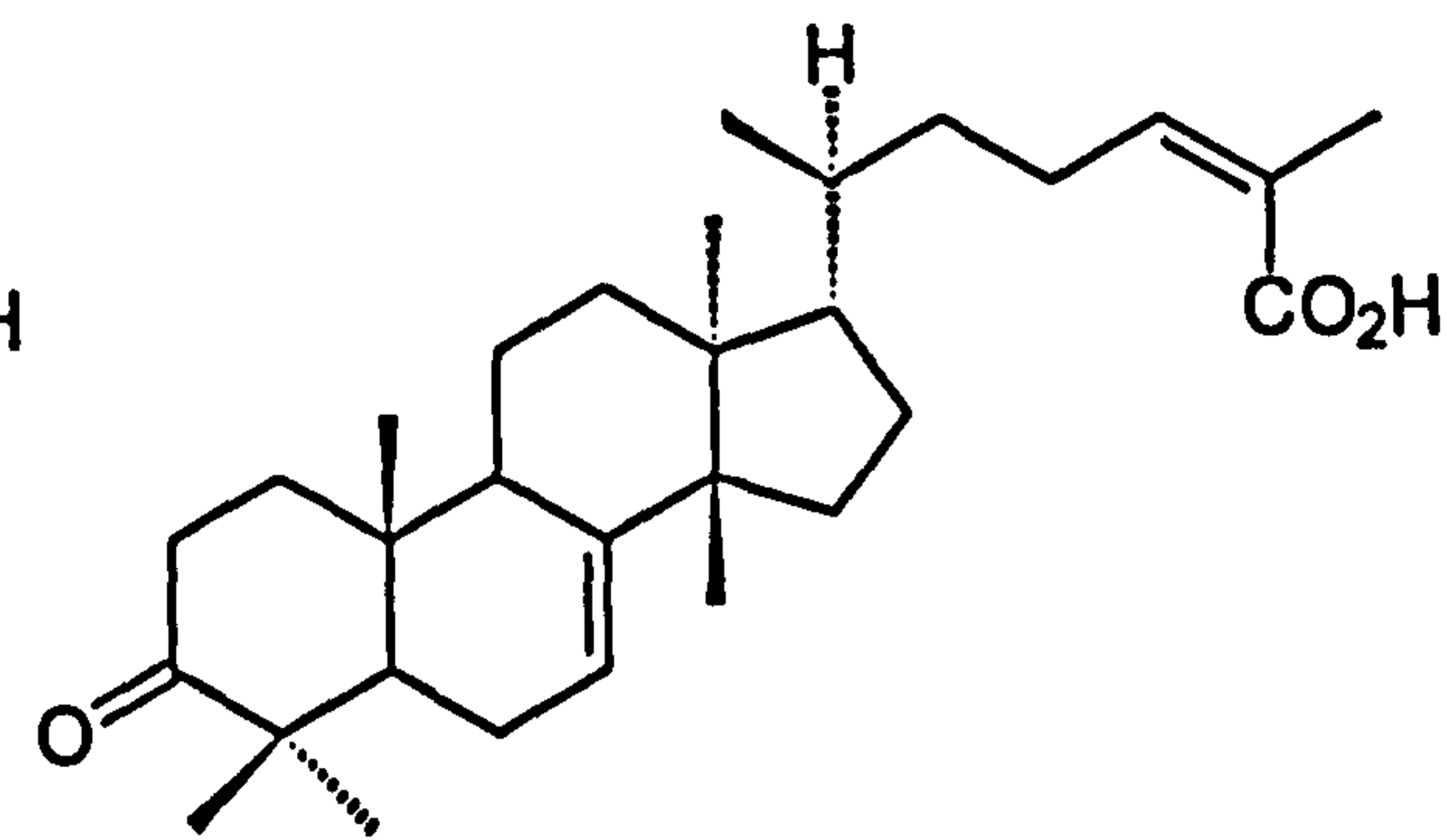
oleanonic acid



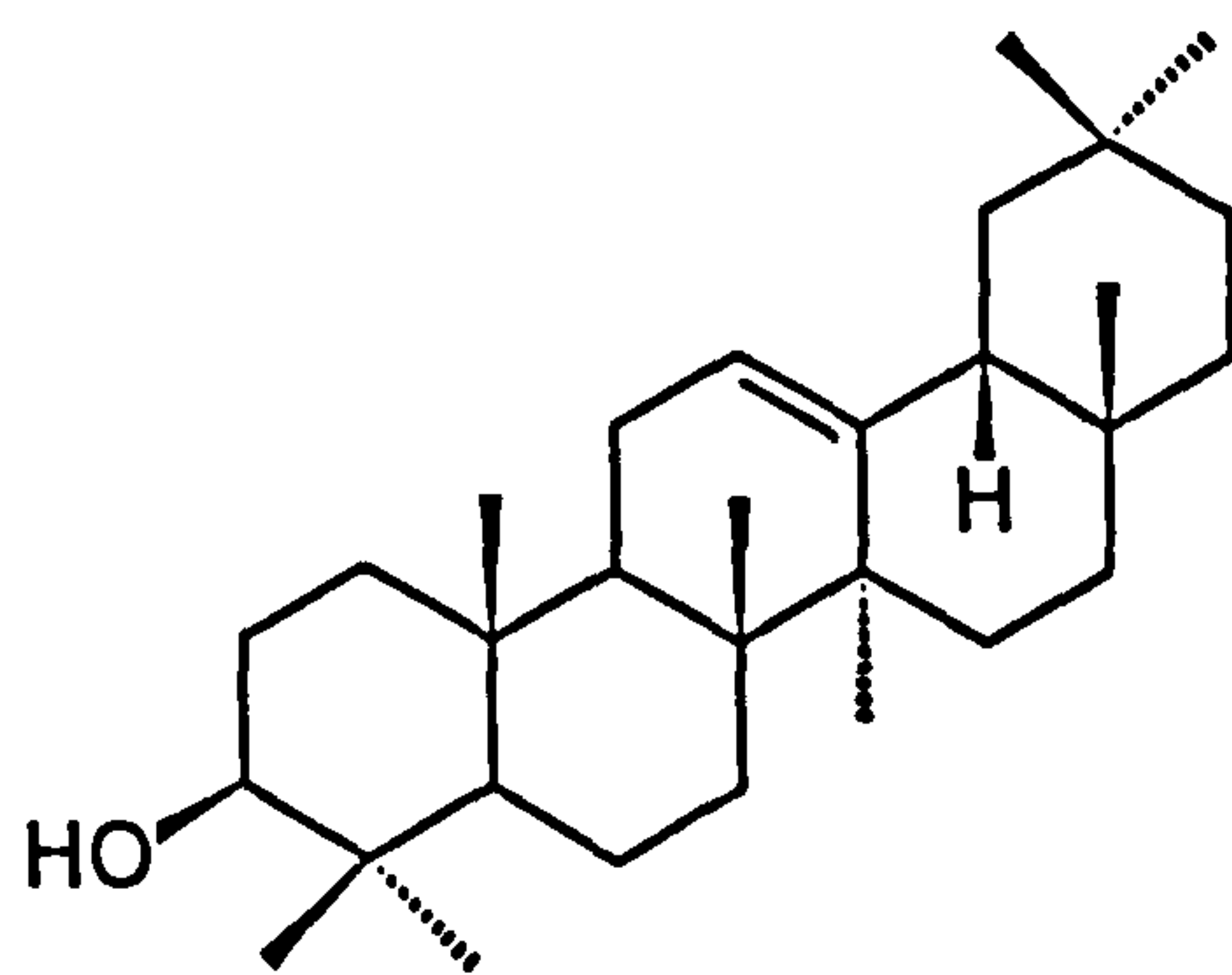
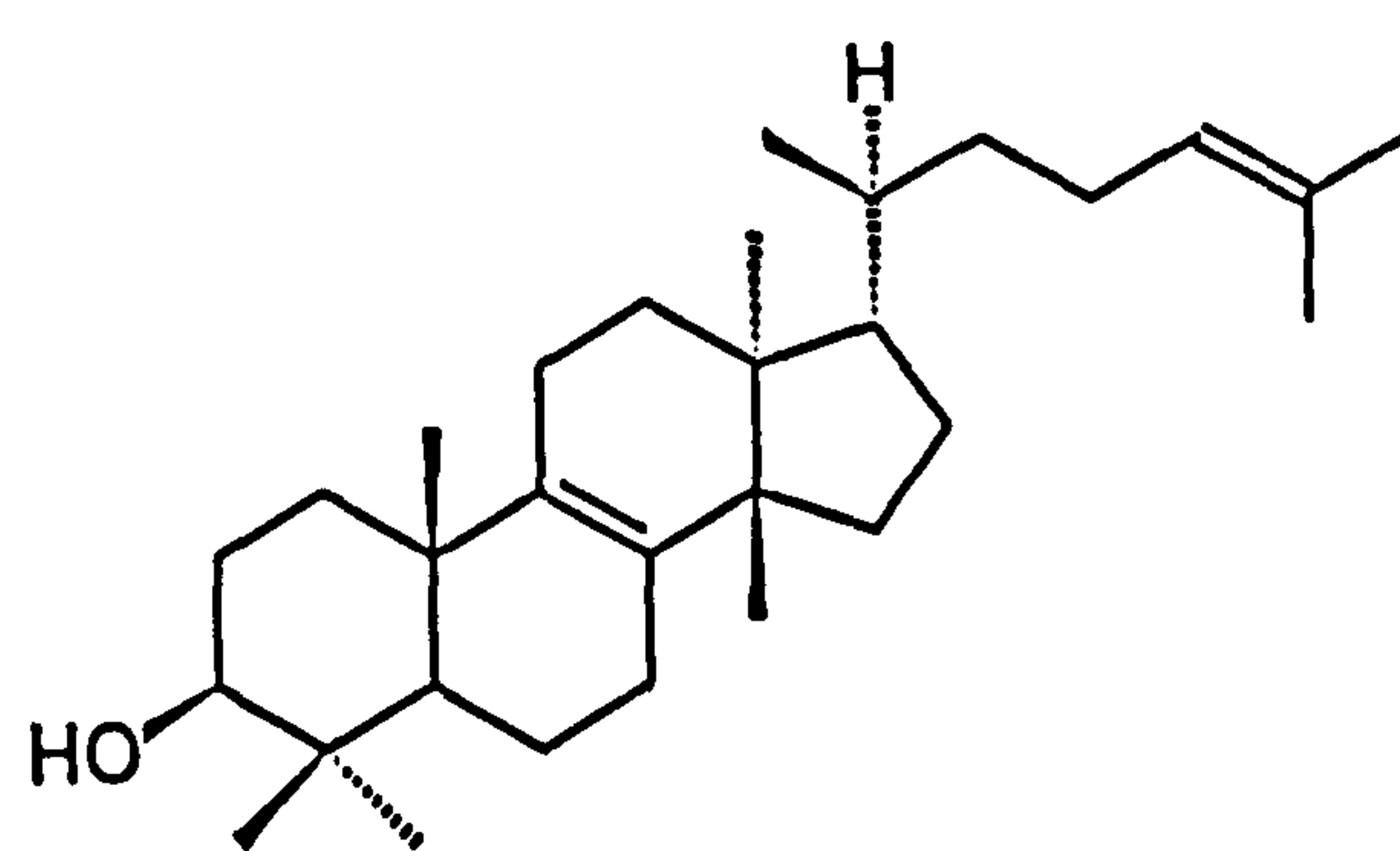
moronic acid



isomasticadienonic acid



masticadienonic acid

 β -amyrin

tirucallol

Figure 2.2 The structures of triterpenoid components occurring in *Pistacia* resins.

differentiate between them, despite the unconvincing results of a study which reported achieving the distinction using TLC (Hairfield & Hairfield 1990, 41A-45A).

2.3.4 Gum resins

In contrast to the conifer and *Pistacia* 'true' resins, the gum resins contain both a resinous portion and a water-soluble gum component. It is their alcohol-soluble resin component which contains the characteristic biomarker compounds allowing identification (see Table 2.1). Of the family *Burseraceae*, the resinous components of both frankincense and myrrh contain characteristic triterpenoid acids, and whilst their sugar (gum) component is of little diagnostic value it can aid identification in certain circumstances (see 2.5). Frankincense (*Boswellia* sp.) typically contains 5-9% essential oils, 65-85% alcohol-soluble resins with the remainder being water-soluble gums (Tucker 1986, p.425-433; Rees 1995, p.9-11). The essential oils consist of mono- and sesquiterpenoids, with p-cymene the most abundant in *B. frereana* (Strappaghetti et al 1982, p.214). However, these lower terpenoids are not exclusive to frankincense and so cannot be diagnostic. The triterpenoids include α - and β -amyrin present in other triterpenoid resins, and the characteristic α - and β -boswellic acids and their acetyl derivatives (Tucker 1986, p.425-433; Khalid 1983, Chapter 10; Pardhy & Bhattacharyya 1978, p.176-178; Hairfield et al. 1989, p.127-133; Bergen et al. 1997, p.8409-8412; Evershed et al. 1997, p.667-668) (see Fig. 2.3).

Also diagnostic of frankincense is 11-oxo- β -boswellic acid and its acetyl derivative which have been identified in *Boswellia* species (Pardhy & Bhattacharyya 1978, p.176-178; Snatzke & Vertesy 1967, p.121-132) (see Fig 2.3). The survival of these characteristic triterpenoid acids has been demonstrated in archaeological samples (Bergen et al. 1997, p.8409-8412; Evershed et al. 1997, p.667-668). The gum of frankincense consists of two polysaccharides, one of galactose and arabinose (1:1), and one of galactose and galacturonic acid (2:1 w/w). The gum component is similar to myrrh (Mills & White 1994, p.77) and so is of little diagnostic value.

Myrrh (*Commiphora* sp.) typically comprises about 2-10% essential oils, 30-60% water-soluble gum and 25-40% alcohol soluble resins (Tucker 1986, p.425-433; Rees 1995, p.9-11). The essential oil of fresh myrrh is dominated by furanosesquiterpenoids (see Fig. 2.3), furanoeudesma-1,3-diene making up ~20% of the oil with significant amounts of curzarene, furanodiene and furanoeudesma-1,4-dien-6-one (Brieskorn & Noble 1983,

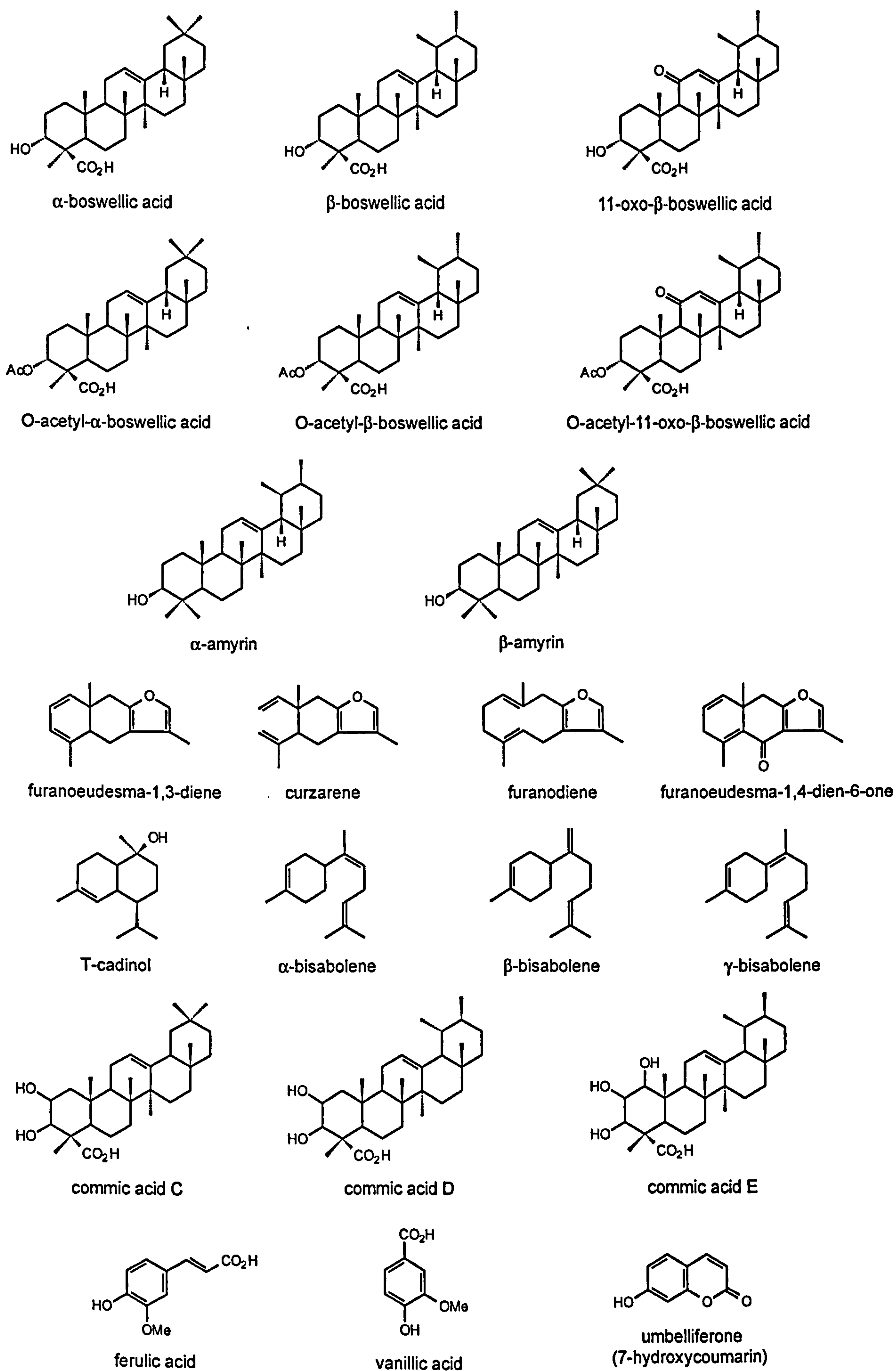


Figure 2.3 The structures of triterpenoid, sesquiterpenoid and aromatic components occurring in gum resins.

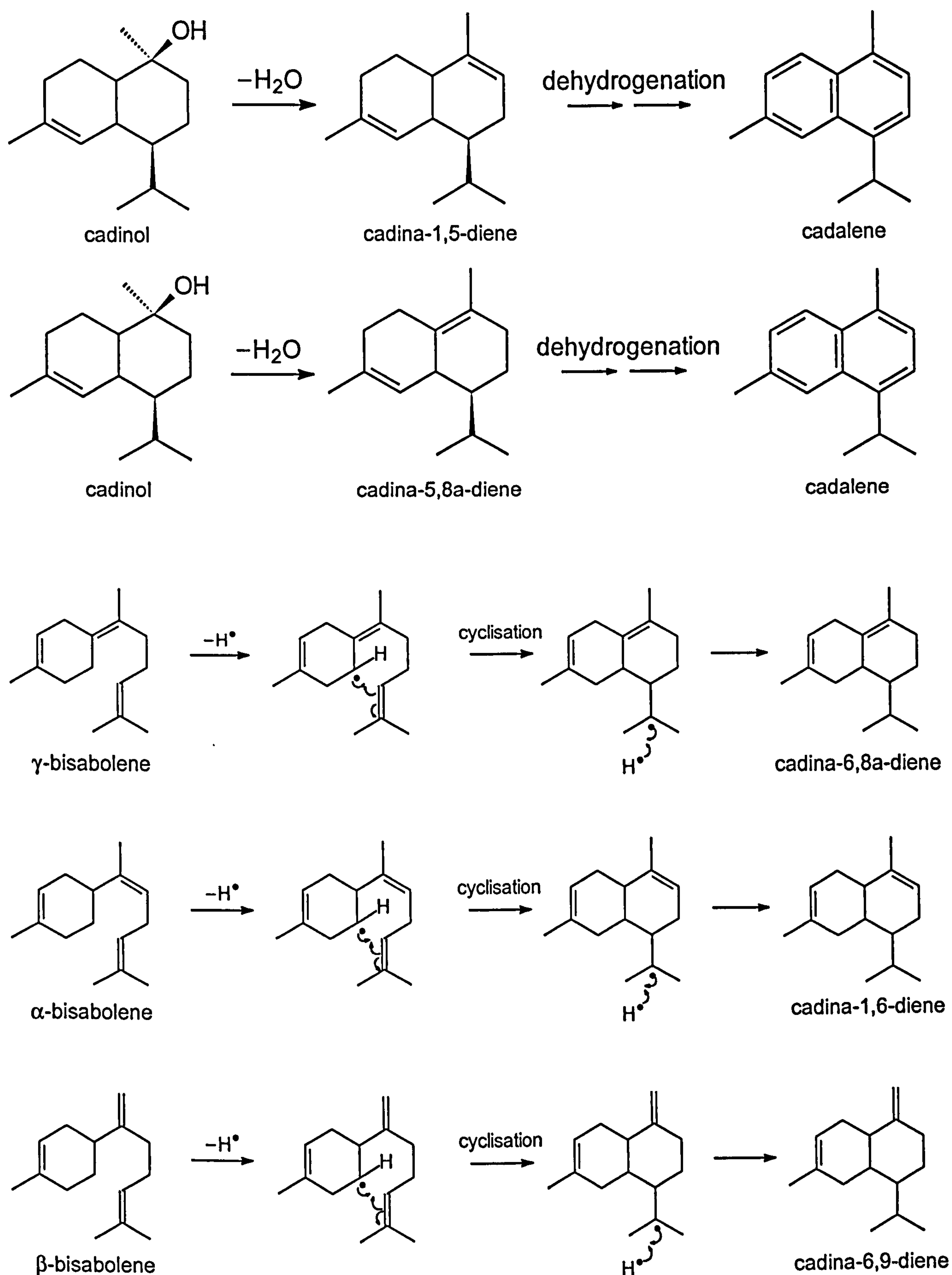


Figure 2.4 Reaction schemes for the conversion of cadinol and bisabolene occurring in myrrh to the less diagnostic cadalene and cadinenes.

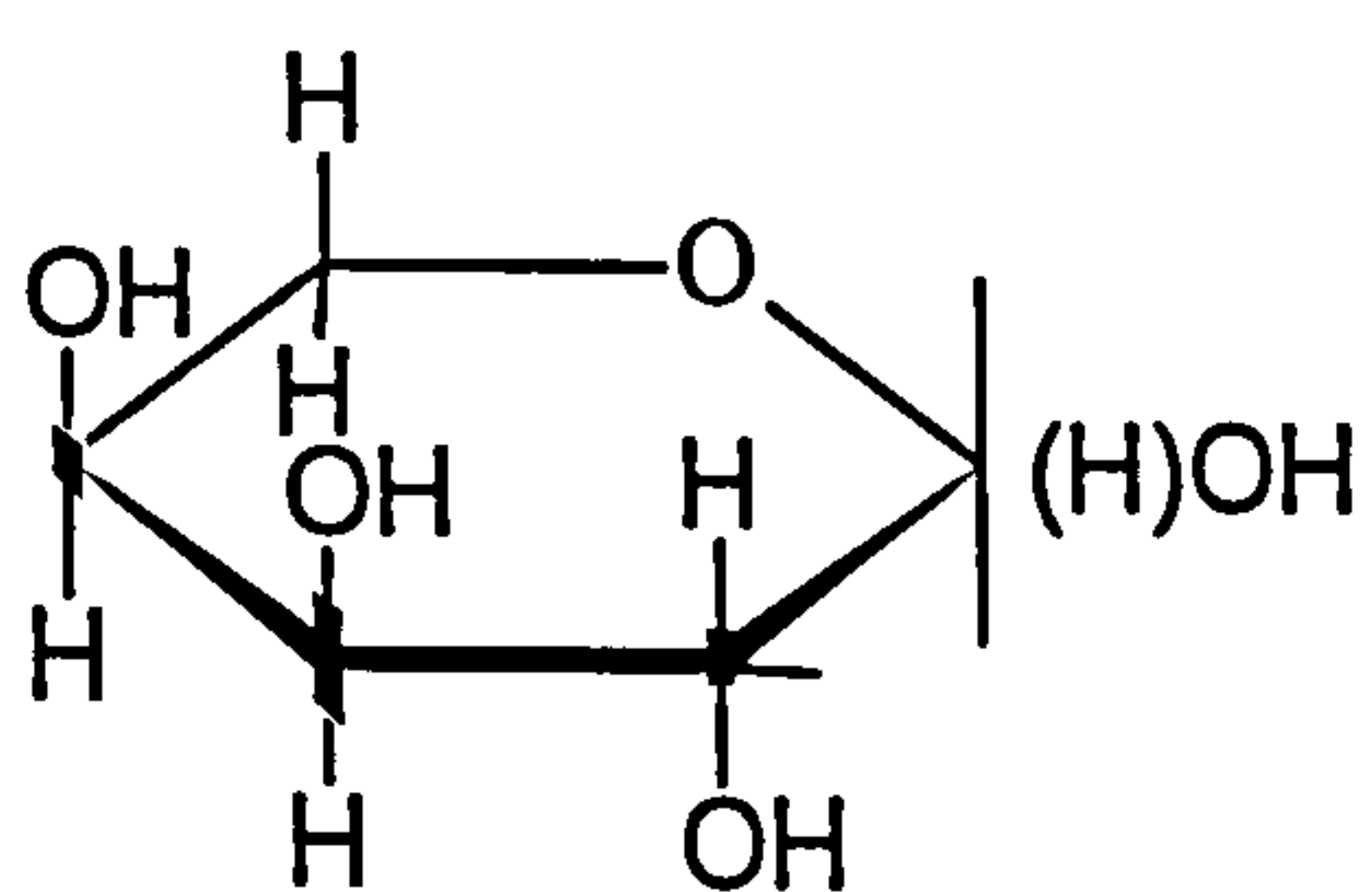
p.187-189; Dolara et al. 1996, p.29; Maradufu & Warthen 1988, p.181-184), although others have been reported (Maradufu 1982, p.677-680; Brieskorn & Noble 1983, p.1207-1211). However, these are highly unstable compounds and are easily autoxidised in air.

T-cadinol and bisabolenes are more stable sesquiterpenoids of the oil component (Manguro et al. 1996, p.84-85; Finar Vol 2, 1975, p.415-416; Andersson et al. 1997, p.250-254), although both can again be converted to cadinenes and cadalene via dehydration and cyclisation, respectively (see Fig. 2.4). The triterpenoids present in myrrh include α -amyrin, β -amyrin and, most importantly, the commic acids (Fig. 2.3) (Thomas & Willhalm 1964, p.3177-3183); these acids being both characteristic of myrrh, and sufficiently stable to be expected to survive in archaeological samples (Thomas & Willhalm 1964, p.3177-3183; Serpico 2000, p.447). The polysaccharides, as mentioned above, are of little value in the identification of myrrh.

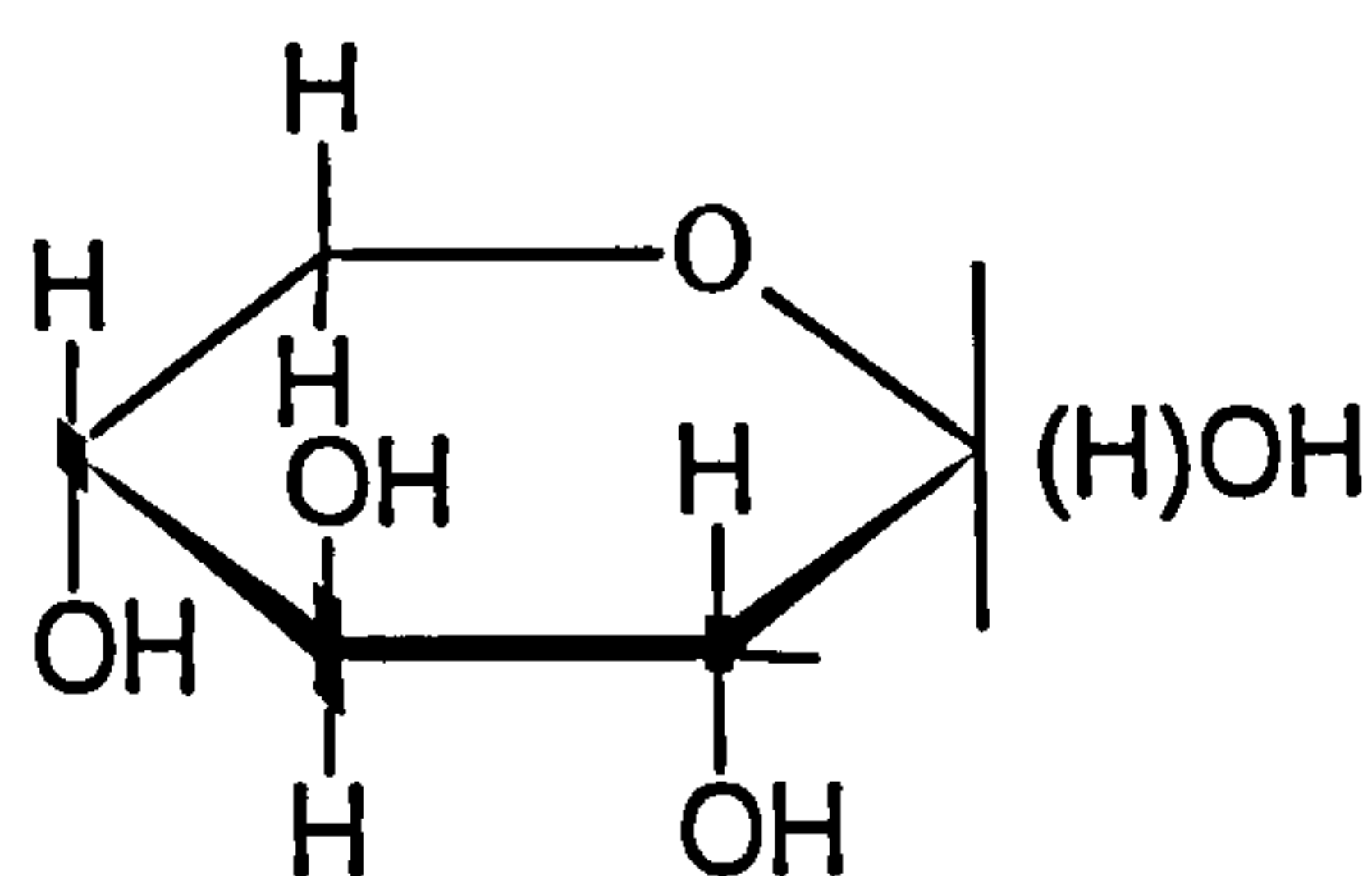
Galbanum is a non-terpenoid resin, its main constituent being ferulic acid which is susceptible to β -oxidation, resulting in the less diagnostic vanillic acid (Fig. 2.3). It also contains phenolic compounds, together with polysaccharide gums which can undergo polymerisation making them less amenable to analysis (Serpico 2000, p.450). However, umbelliferone (7-hydroxycoumarin) (see Fig. 2.3) is a component of galbanum (Benson et al. 1979, p.128), which would be expected to survive in many archaeological environments as indeed identified in an Egyptian mummy (Benson et al. 1979, p.119-131).

2.3.5 Plant gums

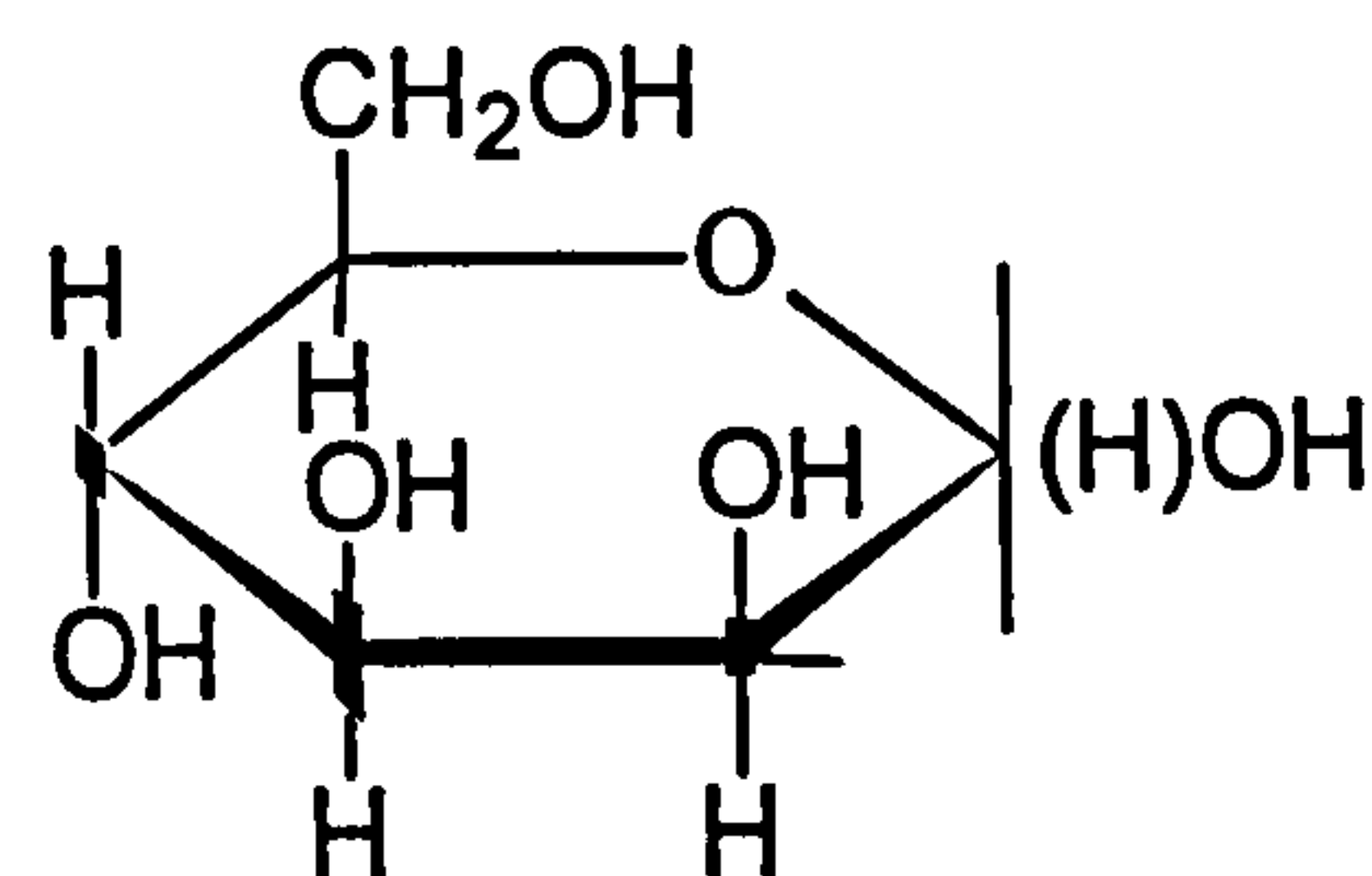
These sugar-based gums largely consist of polysaccharides, made up of a number of sugar and/or uronic acid components (see Table 2.1 and Fig. 2.5). The monosaccharide compositions present in acacia, carob and tragacanth differ sufficiently to be distinguishable from one another, although care would be needed since other sugar-containing materials may have similar monosaccharide distributions. The major sugars in acacia gum are the monosaccharides arabinose and galactose, with lesser amount of rhamnose, glucuronic acid and galacturonic acid (Mills & White 1994, p.77). The acid components in acacia result in the gum existing as a salt with cations of calcium, magnesium and potassium (Mills & White 1994, p.77). It also contains triterpenoid glycosides (Mahato et al. 1992, p.6717, 6728), the triterpenoid component (acacic acid lactone and its derivatives, see Fig. 2.5) of which would be expected to survive and could



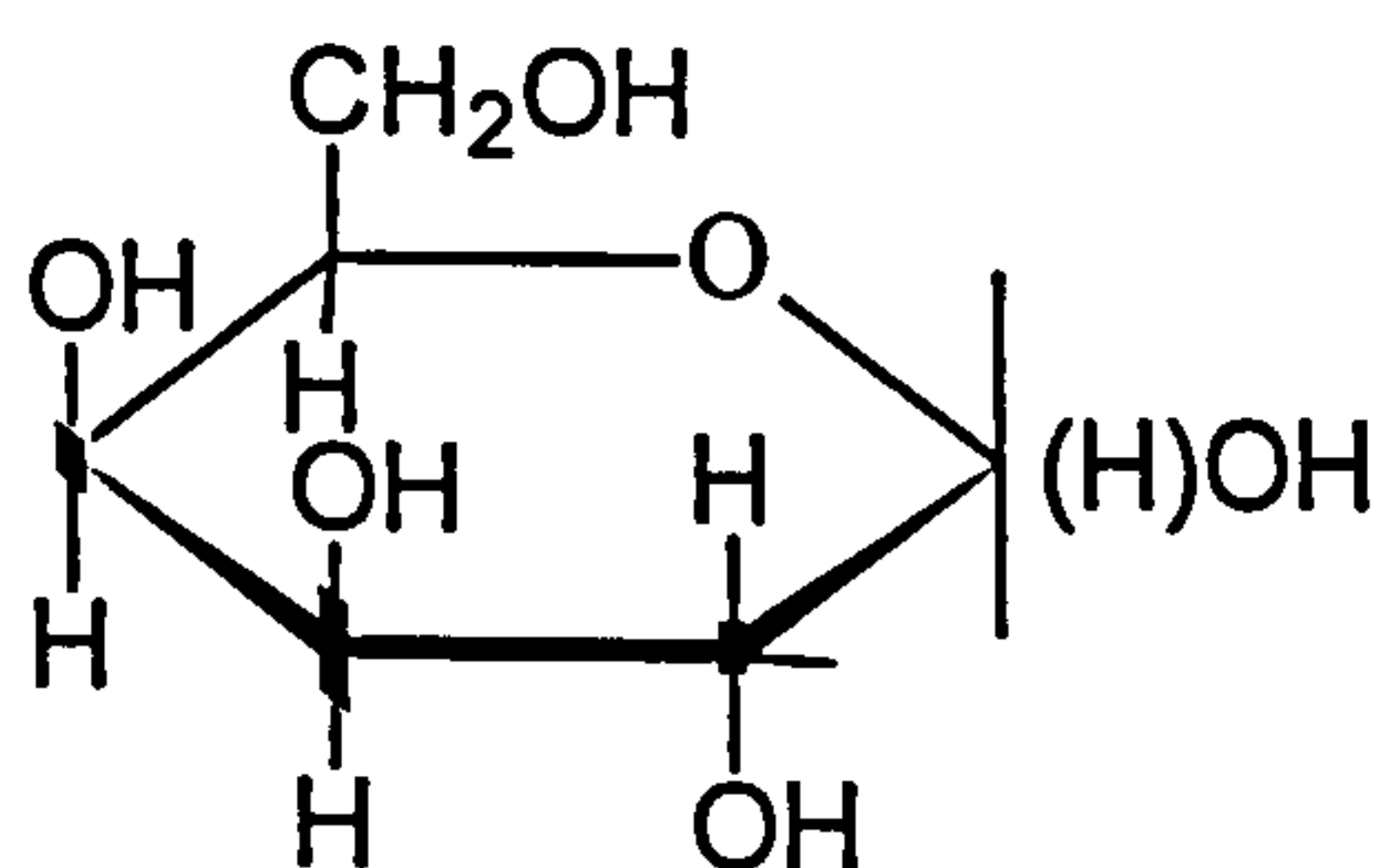
L-arabinose



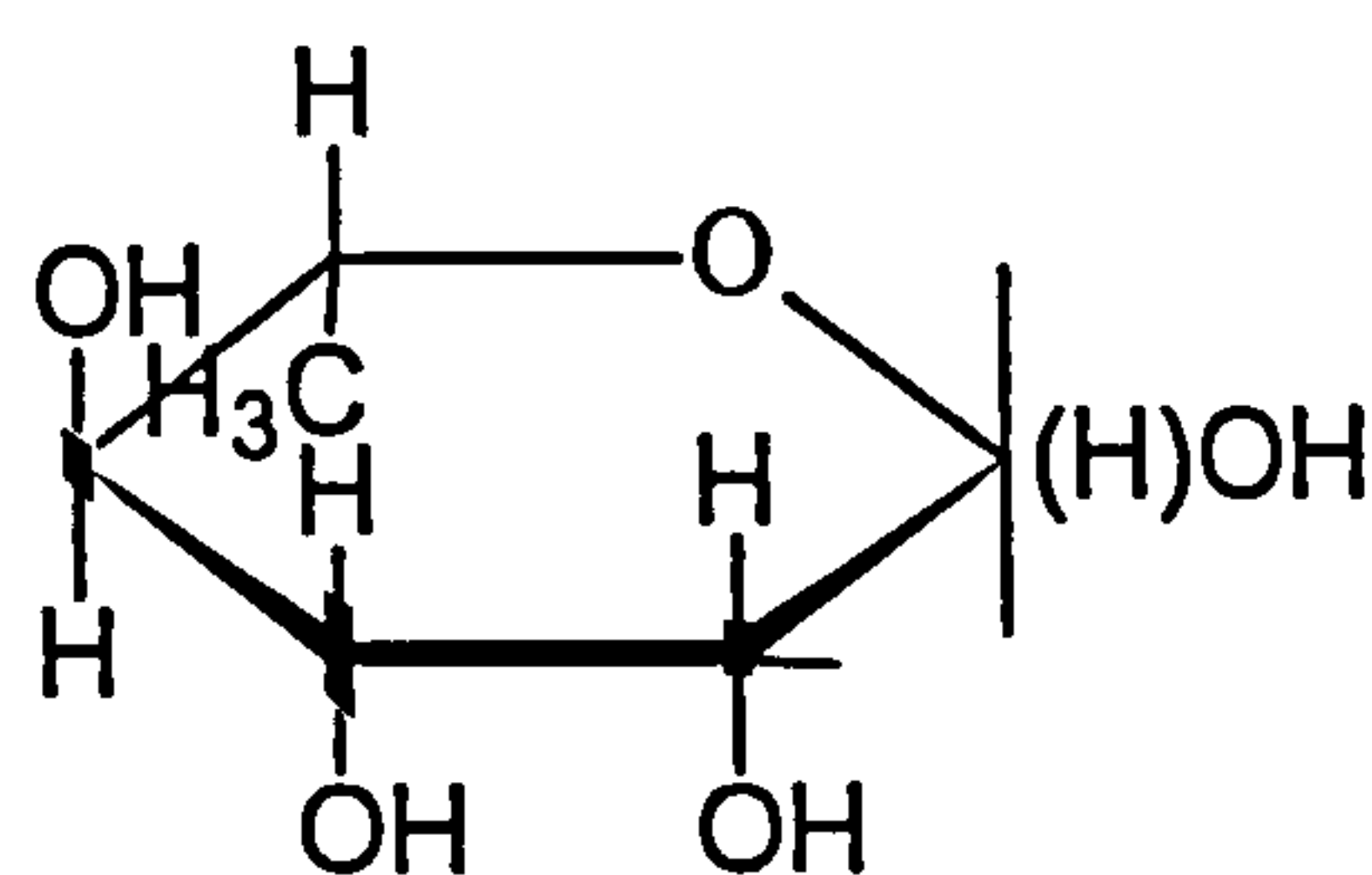
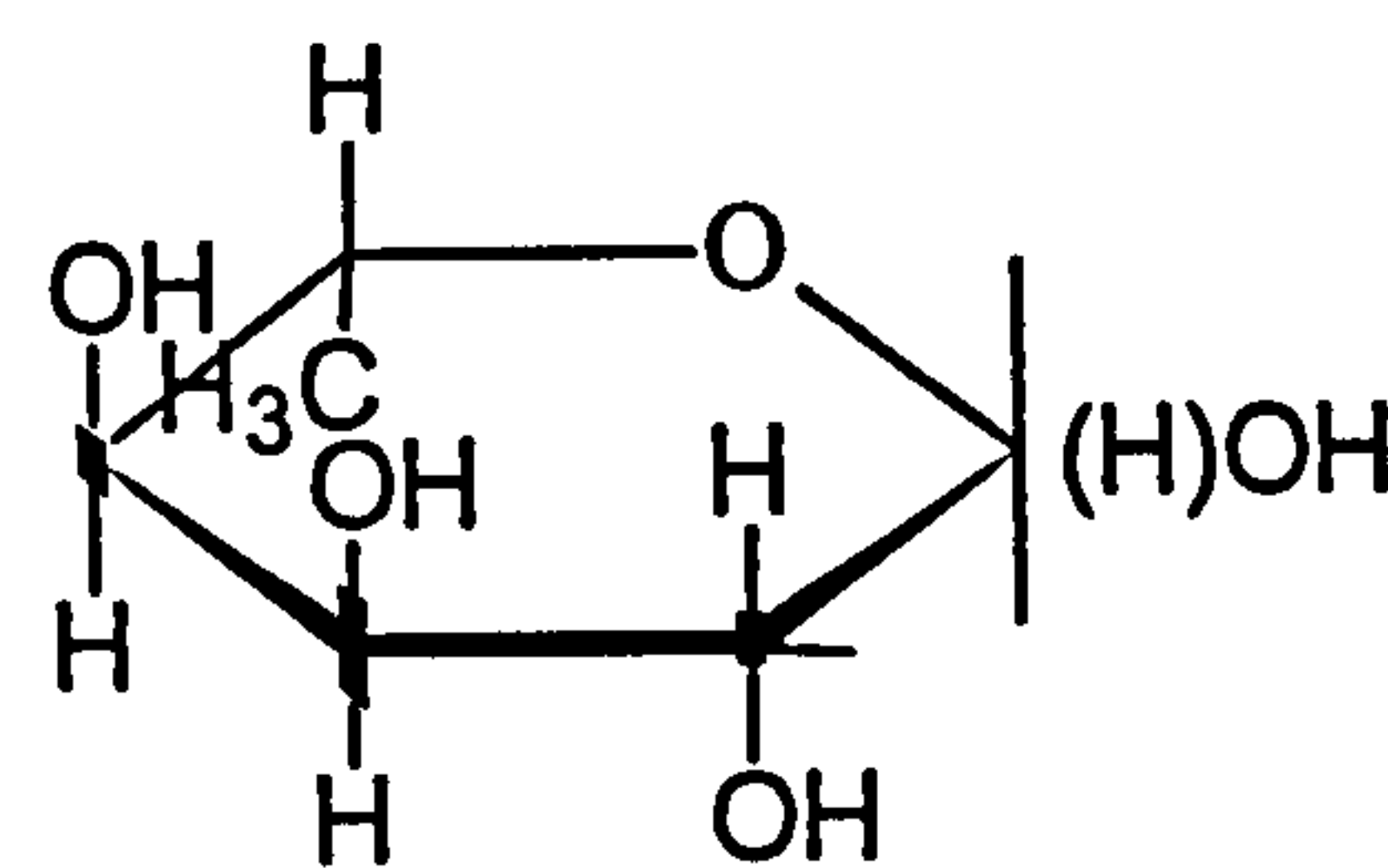
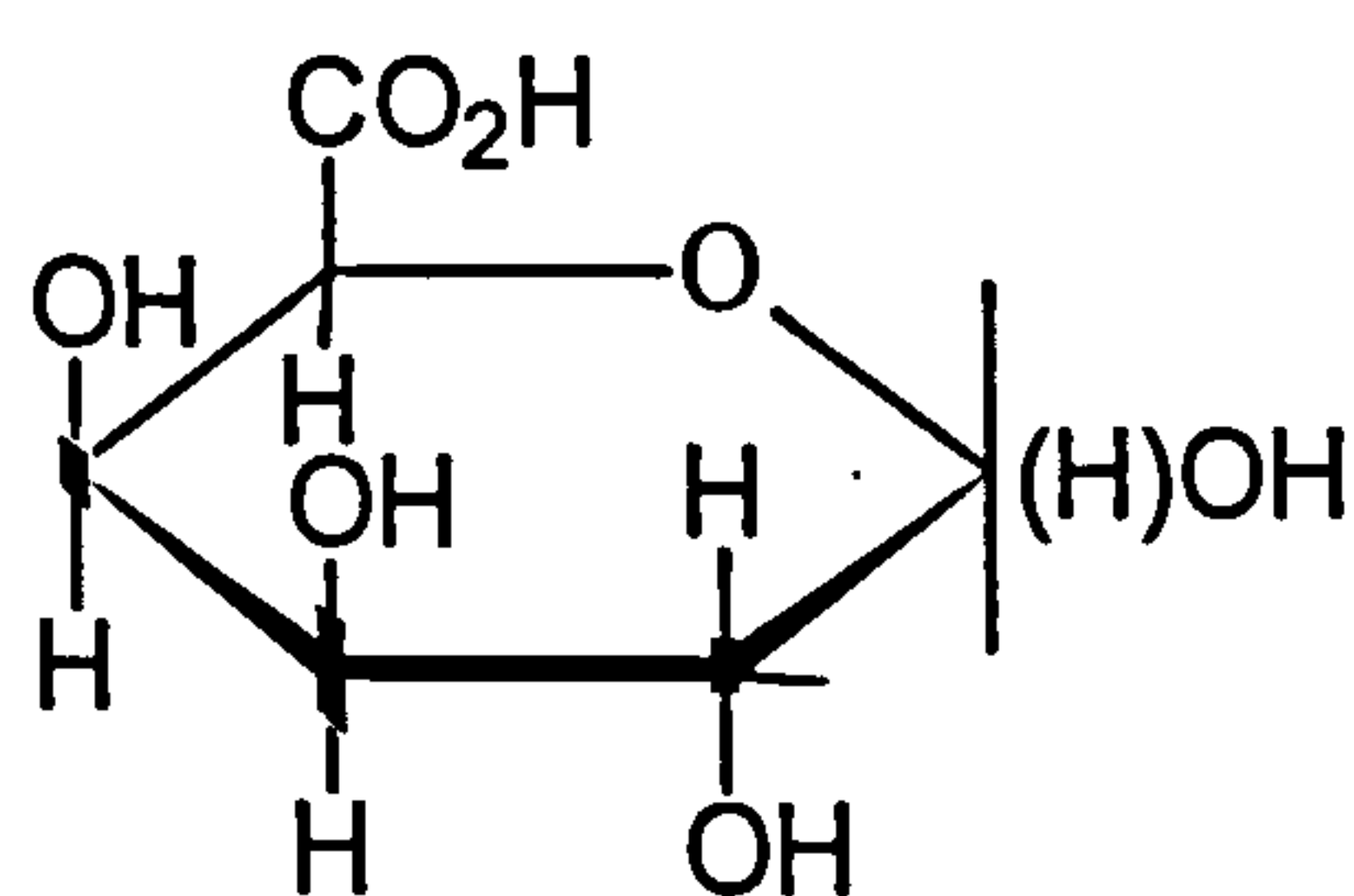
D-xylose



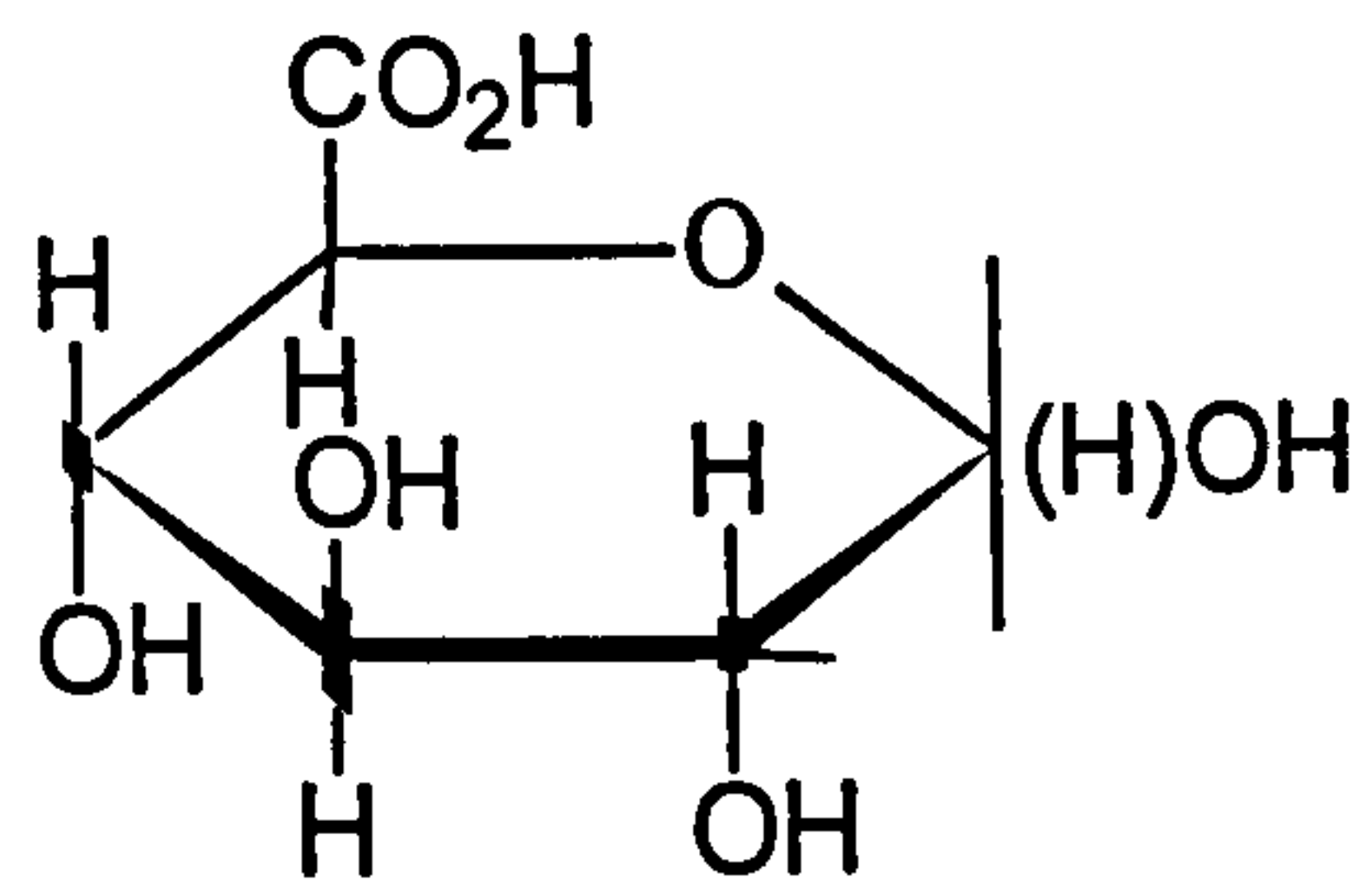
D-mannose



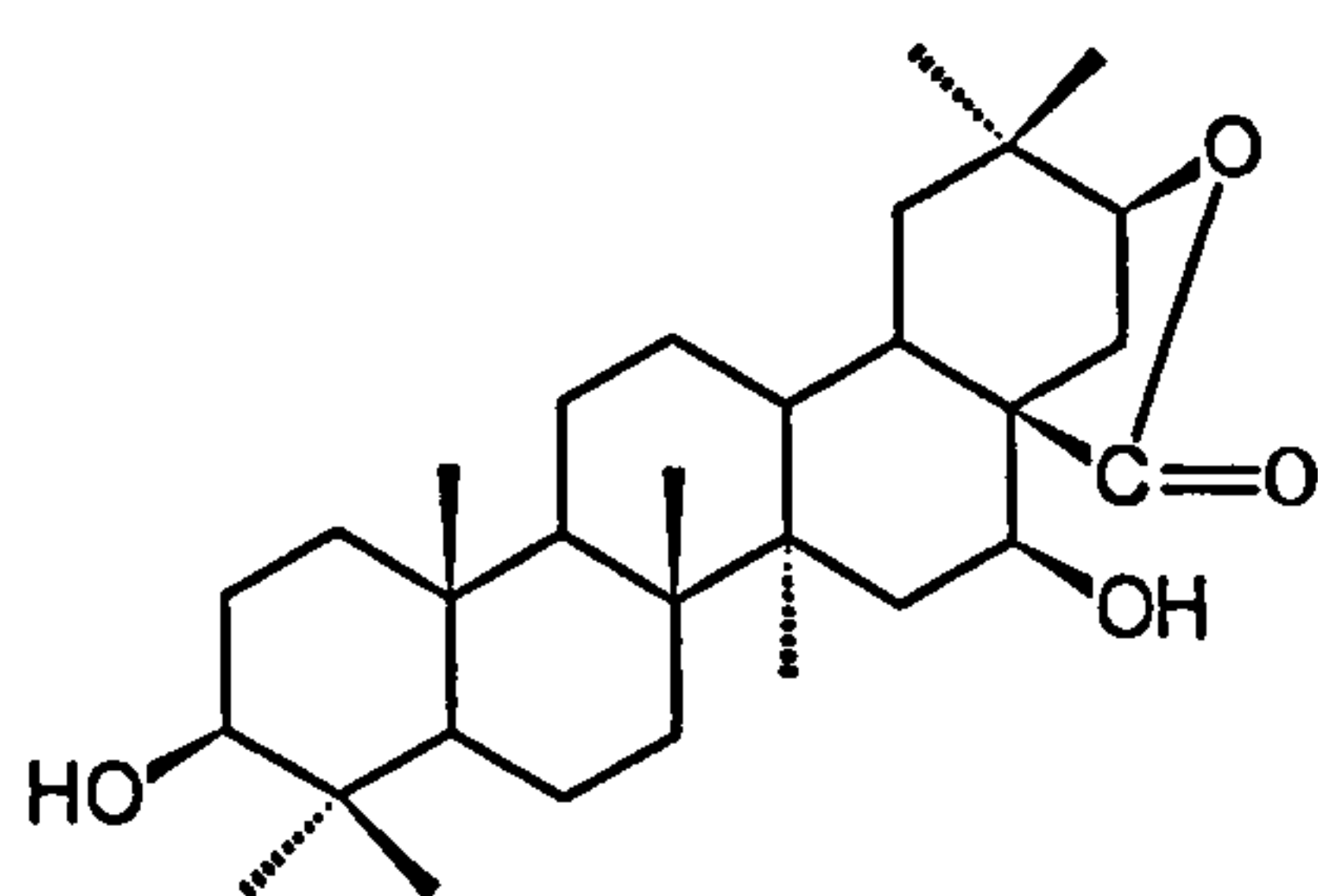
D-galactose

L-rhamnose
(6-deoxy-L-mannose)L-fucose
(6-deoxy-L-galactose)

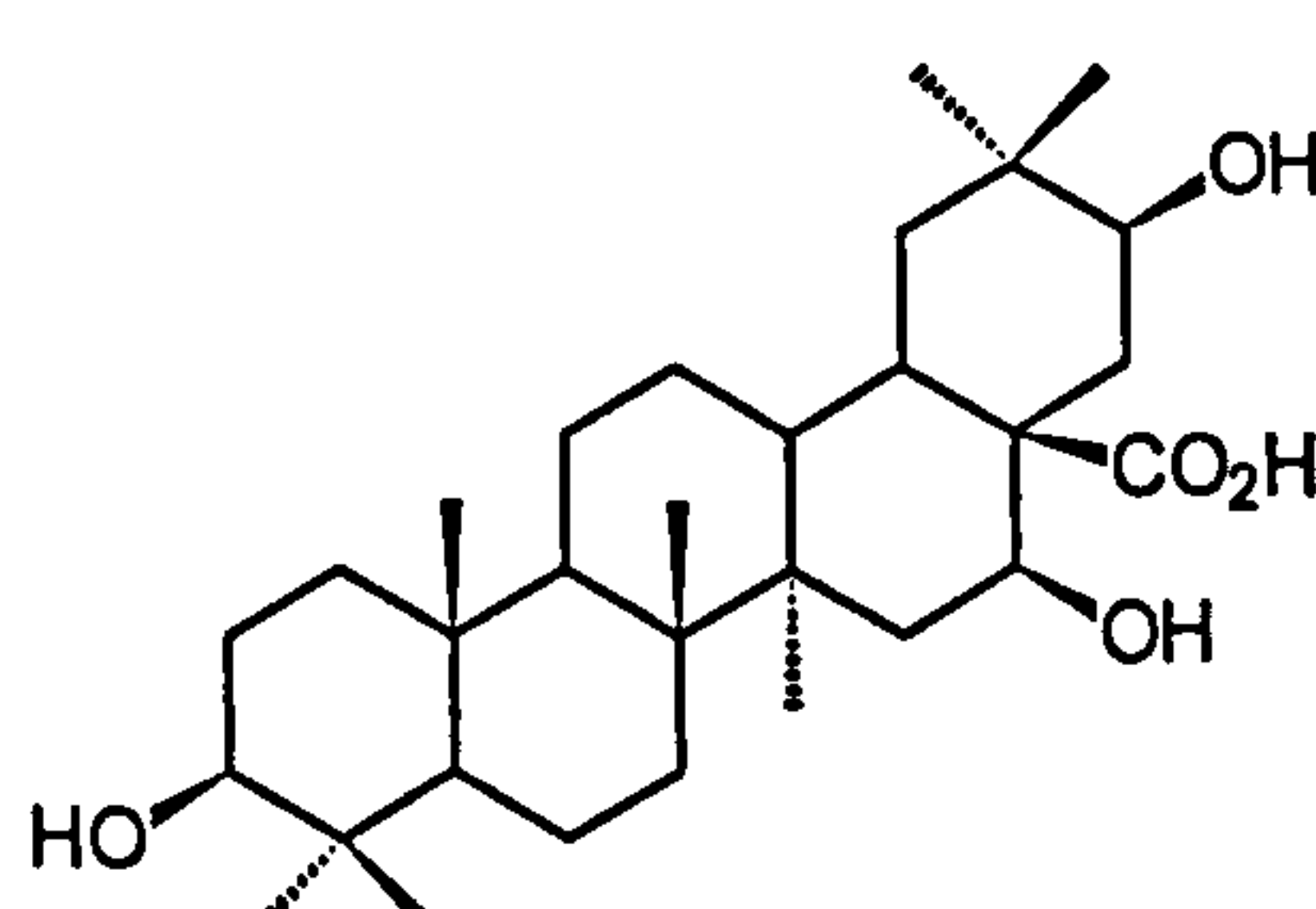
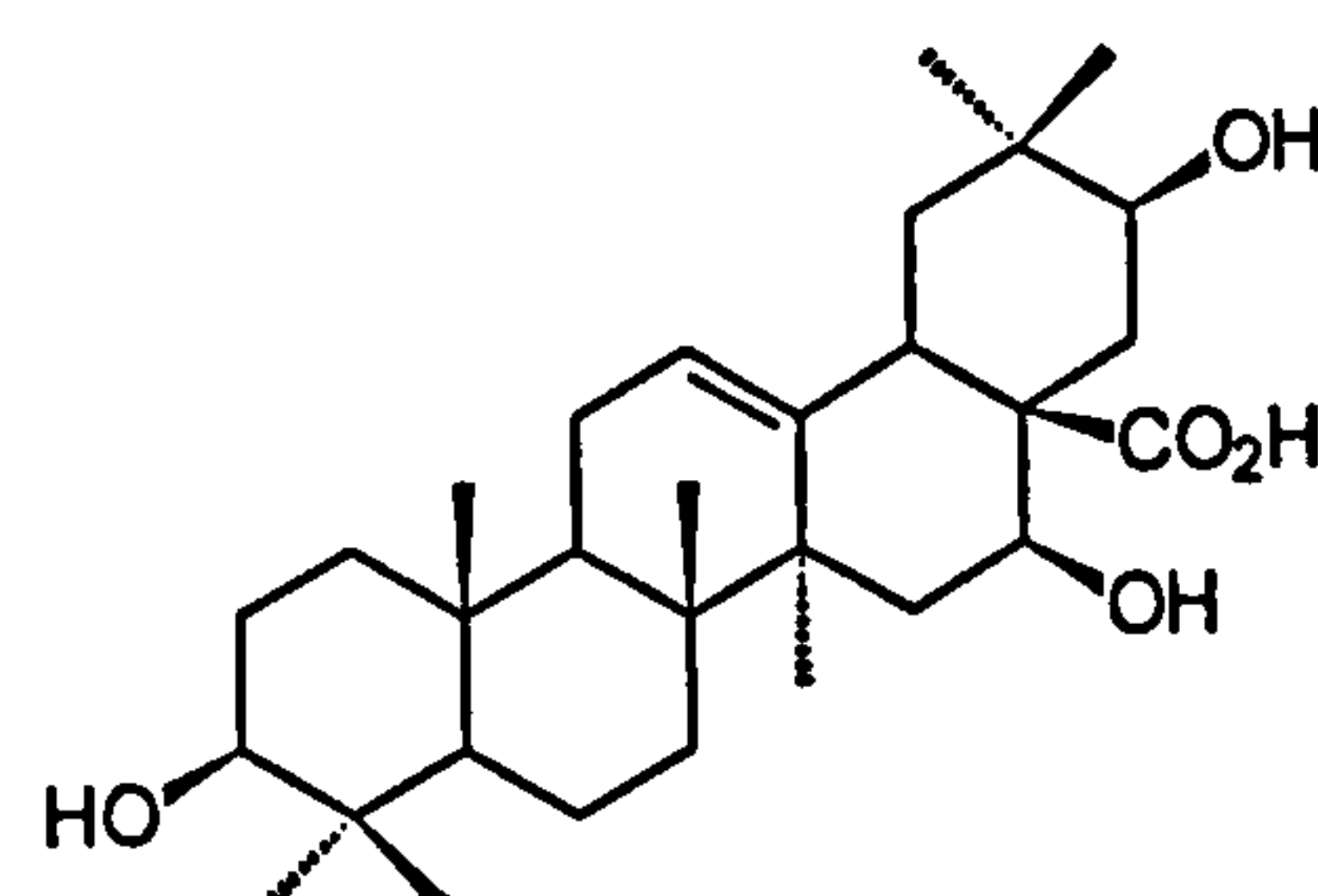
D-galacturonic acid



D-glucuronic acid



acacic acid lactone

16,21-dihydroxy-12,13-dihydro-
oleanolic acid

16,21-dihydroxyoleanolic acid

Figure 2.5 The structures of monosaccharide, uronic acid and triterpenoid components occurring in plant gums. There are two possible anomeric forms (α and β) for each of the monosaccharides and all are shown as their pyranoses.

potentially be used along with the sugars to identify this gum with a reasonable degree of certainty.

In archaeological samples the triterpenoids may be present as dihydroxyoleanolic and dihydroxydihydrooleanolic acids (see Fig 2.5), recognisable given sufficient sample sizes. Carob (*Ceratonia siliqua*) gum contains mannose as the major monosaccharide, with lesser amounts of galactose (Mills & White 1994, p.77). Gum tragacanth (*Astragalus*) contains the sugar components arabinose, galactose, xylose fucose and galacturonic acid, with no single sugar dominant (Mills & White 1994, p.77).

2.3.6 Plant oils (see Table 2.1, Fig 2.6)

The plant oils available to the ancient Egyptians included almond, balanos, castor, colocynth, lettuce, linseed, moringa, olive, poppy, radish, safflower, sesame and tiger nut. Although it is often difficult to distinguish between these various vegetable oils within an archaeological context, certain conclusions are nevertheless possible. The fresh oils consist largely of triglycerides with small amount of phytosterols, particularly β -sitosterol, stigmasterol and campesterol, and lesser amounts of tocopherols, in addition to many other lipids. The fatty acid distribution within the triglycerides of these plant oils can provide useful information regarding the origin of the particular oil, and characterisation of vegetable oils using their fatty acid composition has been carried out using discriminant analysis (Lee et al. 1998, p.163-175).

Yet this approach is problematic with archaeological samples, since the oils will have undergone a marked change in chemical composition (Mills & White 1994 p.34-35; Gulacar et al. 1990, p.691-705). In particular, the polyunsaturated fatty acids abundant in fresh oils are absent, and the monounsaturated fatty acids will be greatly reduced. The unsaturated fatty acids are susceptible to oxidation (Frankel 1982, p.1-33; Frankel et al. 1984, p.2233-2240; Gulaçar et al. 1989, p.61-72; Gulaçar et al. 1990, p.691-705; Evershed 1992, p.253-265; Regert et al 1998, p.2027-2032), producing hydroxy-, oxo- and α,ω -dicarboxylic fatty acids. The triacylglycerols are often completely hydrolysed, with only free fatty acids remaining, and the phytosterols also susceptible to oxidation (Smith 1996, p.453-469) are often absent. Polymerisation via crosslinking of the double bonds in the unsaturated fatty acids can also have taken place (Mills & White 1994, 35-39; Muizebelt & Nielen 1996, p.545-554). The triacylglycerols which do survive will generally contain the



Figure 2.6 The structures of triacylglycerol, sterol and tocopherol components occurring in plant oils. The major fatty acids composing the acyl groups in the triacylglycerols are also shown as they are often dominant in archaeological samples. Although they do not usually occur in fresh plant oils, the monohydroxy-, dihydroxy- and α,ω -di- fatty acids are often major extractable components in aged archaeological samples. With the exception of oleic and erucic acids the stereochemistry of the unsaturated fatty acids is not shown.

more stable fatty acids and are therefore not necessarily representative of the original oil.

Monoacylglycerols, which often survive in archaeological samples, and result from the partial hydrolysis of triacylglycerols, are dominated by the saturated $C_{16:0}$ and $C_{18:0}$ fatty acids which are again of little diagnostic value. Given the highly degraded nature of most archaeological oil samples, the major acid remaining is generally palmitic acid with smaller amounts of myristic, oleic and stearic acids. Yet utilising the $C_{16:0}/C_{18:0}$ ratio to determine their origin, a ratio of 2:1 or more can generally indicate a vegetable oil rather than an animal derived fat (Serpico & White 1996, p.128-139).

Despite the difficulties of providing more than a general identification of plant oil, it may be possible to be more specific when examining a particular oil in which more unusual fatty acids are present. Castor is one such oil and consists of large amounts of (83-89%) ricinoleic acid (Mills & White 1994, p.33). This can survive as the acid itself or as the 9,12-dihydroxyoctadecanoic acid derivative. Moringa oil contains about 4% long chain fatty acids, including $C_{20:0}$, $C_{22:0}$ and $C_{26:0}$ (Serpico & White 2000, p.414; Somali et al. 1984, p.85-86; Tsaknis 1999, p.4495-4499) which should persist as indicative of moringa. Radish oil contains about 35% erucic acid and 10% arachidic acid (Serpico & White 2000, p.414), the former susceptible to degradation although some is likely to survive along with the more stable $C_{20:0}$ fatty acid facilitating its identification. Tiger nuts contain relatively large amounts (~30%, Eteshola & Oraedu 1996, p.255-257) of myristic acid, which again may well be recognisable.

Palm wine produced from either the tree sap or the fruit (see Chapter 2) may contain fatty acids as minor components, whichever is the source. Palm oil contains a large amount of palmitic acid likely to survive along with smaller amounts of other saturated acids ($C_{14:0}$ and $C_{18:0}$) (Gunstone et al. 1986, p.98-99; McCance & Widdowson 1998, p.79), yet in oil obtained from the fruit lauric acid is the major fatty acid, with smaller but significant amounts of myristic acid and a relatively small abundance of palmitic acid (Gunstone et al. 1986, p.98-99; Copley et al. in press).

The ubiquitous nature of fatty acids (oils, fats, plant waxes, etc.) means that their significance is often uncertain and their interpretation requires great care. Human adipose tissue also contains a relatively high ratio of $C_{16:0}/C_{18:0}$ fatty acids, which must obviously be taken into account when examining material from mummified bodies (Gulaçar et al.

1989, p.61-72; Gulaçar et al. 1990, p.691-705; Evershed 1992, p.253-265) (for a fuller account of the processes involved in fat and oil degradation in an archaeological context see Chapter 4).

2.3.7 Animal fats

Animal fats like plant oils are dominated by triacylglycerols, with steroids present as minor components, cholesterol and its esters the most significant (see Table 2.1 and Fig. 2.7). The triacylglycerols of animal fats differ from plant oils in that they contain more of the saturated fatty acids and consequently are solid at room temperature. The ratio of C_{16:0}/C_{18:0} fatty acids is closer to 1:1 in many animal fats (Mills & White 1994, p.33; Serpico & White 1996, p.128-139; Serpico & White 2000, p.418), although it should be noted that human and pig adipose fat has a relatively high C_{16:0}/C_{18:0} ratio (Mills & White 1994, p.33; Hirsch 1965, p.183; Gellhorn & Benjamin 1965, p.661). The ratio of C_{16:0}/C_{18:0} has been used in various archaeological contexts to identify animal fats and distinguish them from plant oils (Charters et al. 1995, p.113-127; Mottram et al. 1999, p.209-221; Serpico & White 1996, p.128-139).

Ruminant fats (sheep, cattle, goats, etc.) contain significant quantities of branched chain fatty acids and a complex mixture of C_{18:1} fatty acid isomers ($\Delta^{9,10,11,13,14,15,16}$) resulting from the biohydrogenation of unsaturated dietary fatty acids in the rumen (Mottram et al. 1999, p.209-221). These unusual fatty acids can be used to identify a ruminant fat and distinguish it from non-ruminant fats which contain only the C_{18:1} Δ^9 isomer. Stable isotopes can also be used to distinguish between ruminant and non-ruminant fat but in the context of organic embalming agent this approach can be problematic (see Chapter 3). Although often absent in archaeological fats due to degradative processes, steroids can survive in their original form or as degraded but recognisable steroids which include cholesta-3,5-dien-7-one and 7-ketocholesterol (see Fig. 2.7) (Buckley et al. 1999, p.443-452).

2.3.8 Waxes

Beeswax, a mixture of hydrophobic lipids (see Fig. 2.8), is believed to be the only wax available to the ancient Egyptians. It contains C₂₃-C₃₅ hydrocarbons with the C₂₇ n-alkane the most abundant. The C₃₁ hydrocarbon contains a proportion of alkene and the C₃₃ is

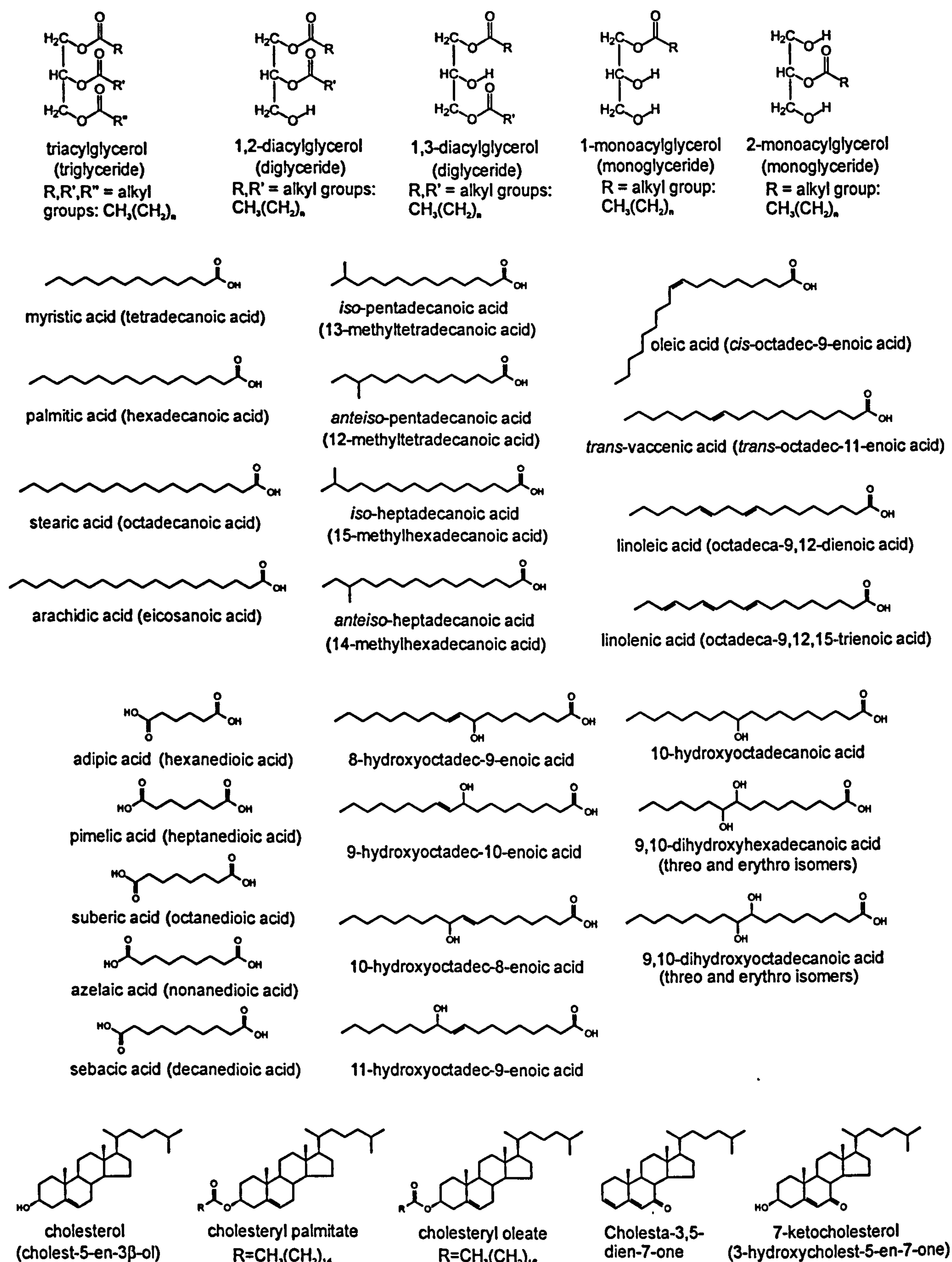


Figure 2.7 The structures of triacylglycerol and sterol components occurring in animal fats. The major fatty acids composing the acyl groups in the triacylglycerols are also shown as they are often dominant in archaeological samples. Although they do not usually occur in fresh animal fats, the monohydroxy-, dihydroxy- and α,ω -di- fatty acids are often major extractable components in aged archaeological samples. With the exception of oleic and vaccenic acids the stereochemistry of the unsaturated fatty acids is not shown.

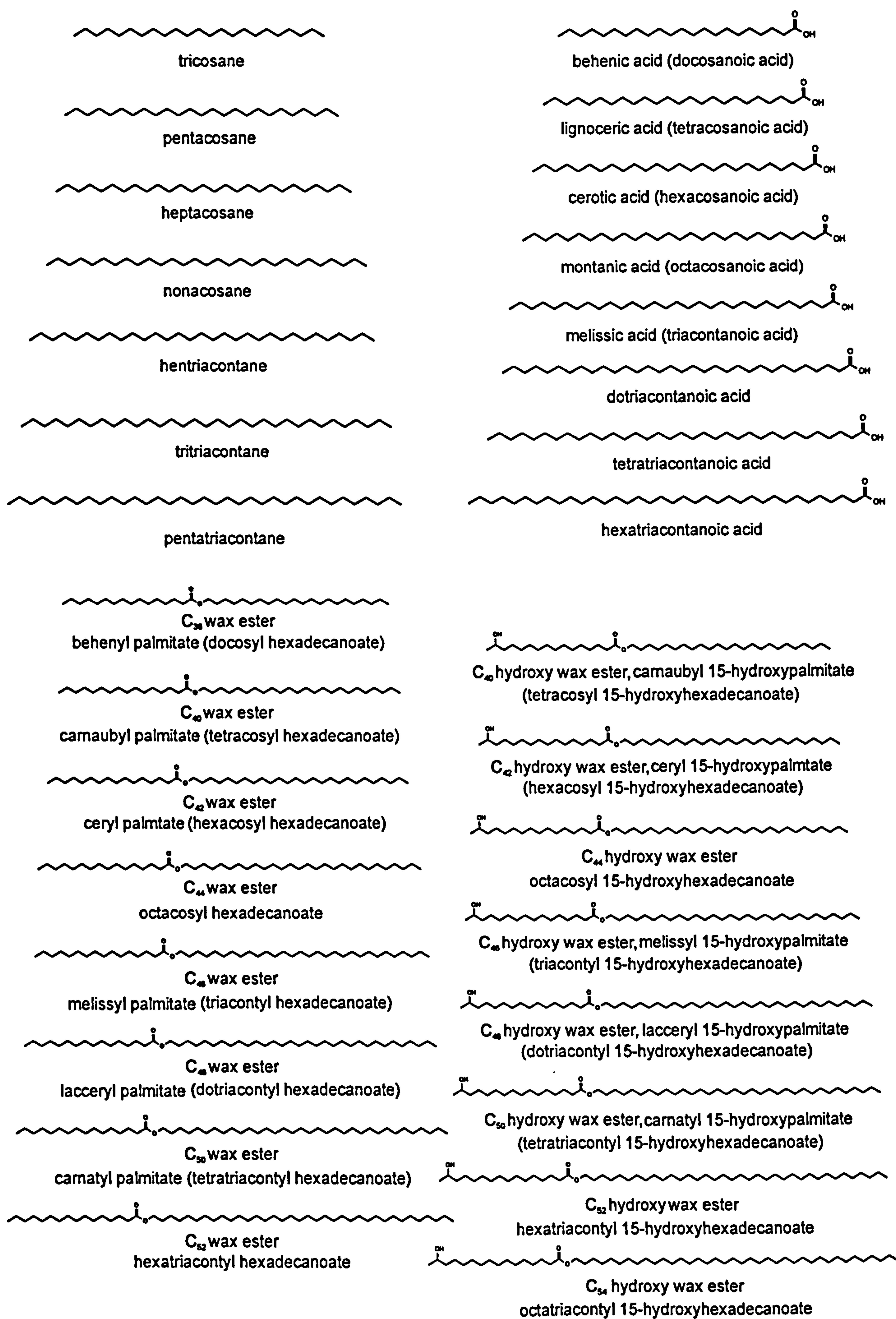


Figure 2.8 The structures of alkane, free fatty acid, wax ester and hydroxy wax ester components occurring in beeswax.

present largely as the alkene in fresh beeswax whereas in archaeological samples the alkenes are absent (Mills & White 1994, p.50). The fresh wax also contains C₃₈-C₅₂ wax monoesters (and di- and triesters) of palmitic acid, with smaller amounts of oleic, stearic and arachidic acids and straight chain alcohols (C₂₂-C₃₆) (Tulloch 1971, p.235-265).

The presence of predominantly palmitic acid is indicative of beeswax, since the wax esters in other forms of wax tend to be mixtures of longer chain fatty acids (Tulloch 1973, p.367-371). It should be said, however, that in certain archaeological environments these beeswax esters may undergo transesterification (Bull et al. 1998, p.11-26; Jambu et al. 1995, p.187-192), with longer (or shorter) chain fatty acids replacing the C_{16:0} fatty acid moiety. Particularly diagnostic of beeswax are the hydroxy wax esters (C₄₀-C₅₄) and with the exception of palmitic acid, lignoceric acid is often the dominant fatty acid present. Due to their lack of volatility and their relative inertness, most of the components should survive as diagnostic biomarkers, as indeed they have in a number of archaeological contexts (Charters et al. 1995, p.113-127; Heron et al. 1994, p.266-269; Connan & Dessort 1991, p.1445-52, Evershed et al. 1997, etc. see previous investigations).

2.3.9 Spices

The essential oils of cassia (*Cinnamomum cassia*, *Cassia* spp.) and cinnamon (*Cinnamomum zeylanicum*) have a similar chemistry (Miller et al. 1995, p.461-471), the major component in both being *trans*-cinnamaldehyde together with lesser amounts of terpenes, aromatic compounds (see Fig. 2.9) and esters (see Fig. 2.9). Yet they are distinguishable, since cassia contains the components coumarin and δ -cadinene absent in cinnamon whilst cinnamon contains the minor components eugenol and benzyl benzoate which are either absent in cassia or only present as a trace (Miller et al. 1995, p.461-471).

Although these volatile molecules may no longer be present in archaeological samples, their importance merits their inclusion. It is also remotely possible that they could be trapped in the sample matrix (e.g. resin), in which case benzoic, cinnamic and hydroxyaromatic acids may be present along with δ -cadinene, and coumarin and eugenol derivatives.

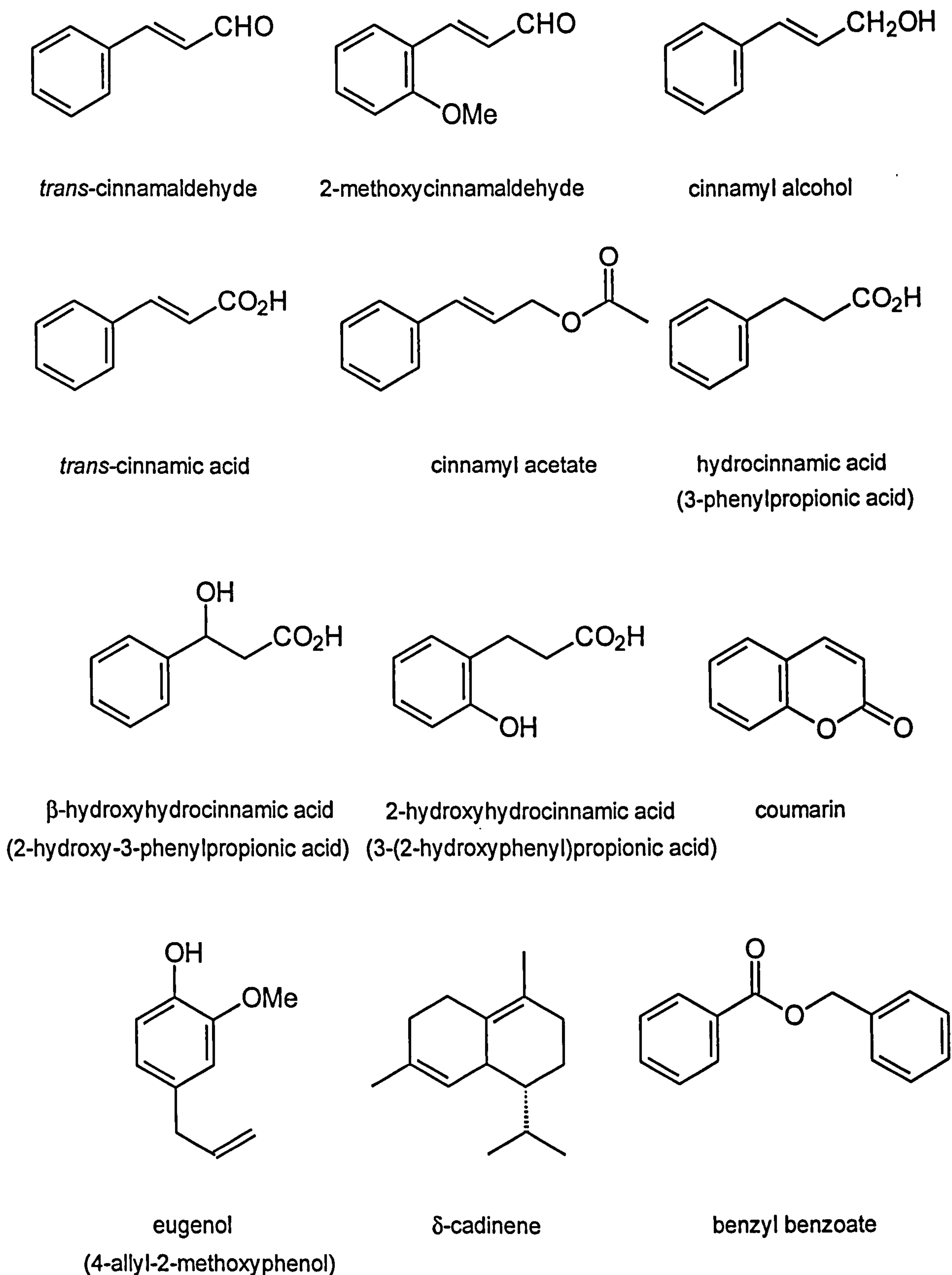


Figure 2.9 The structures of aromatic and sesquiterpenoid components occurring in the spices cassia and cinnamon. Although they do not occur in the fresh spices, hydrocinnamic acid and hydroxyhydrocinnamic acids are possible degradation products which could be expected to be observed in archaeological samples if cassia or cinnamon were present.

2.3.10 Vegetable dyes

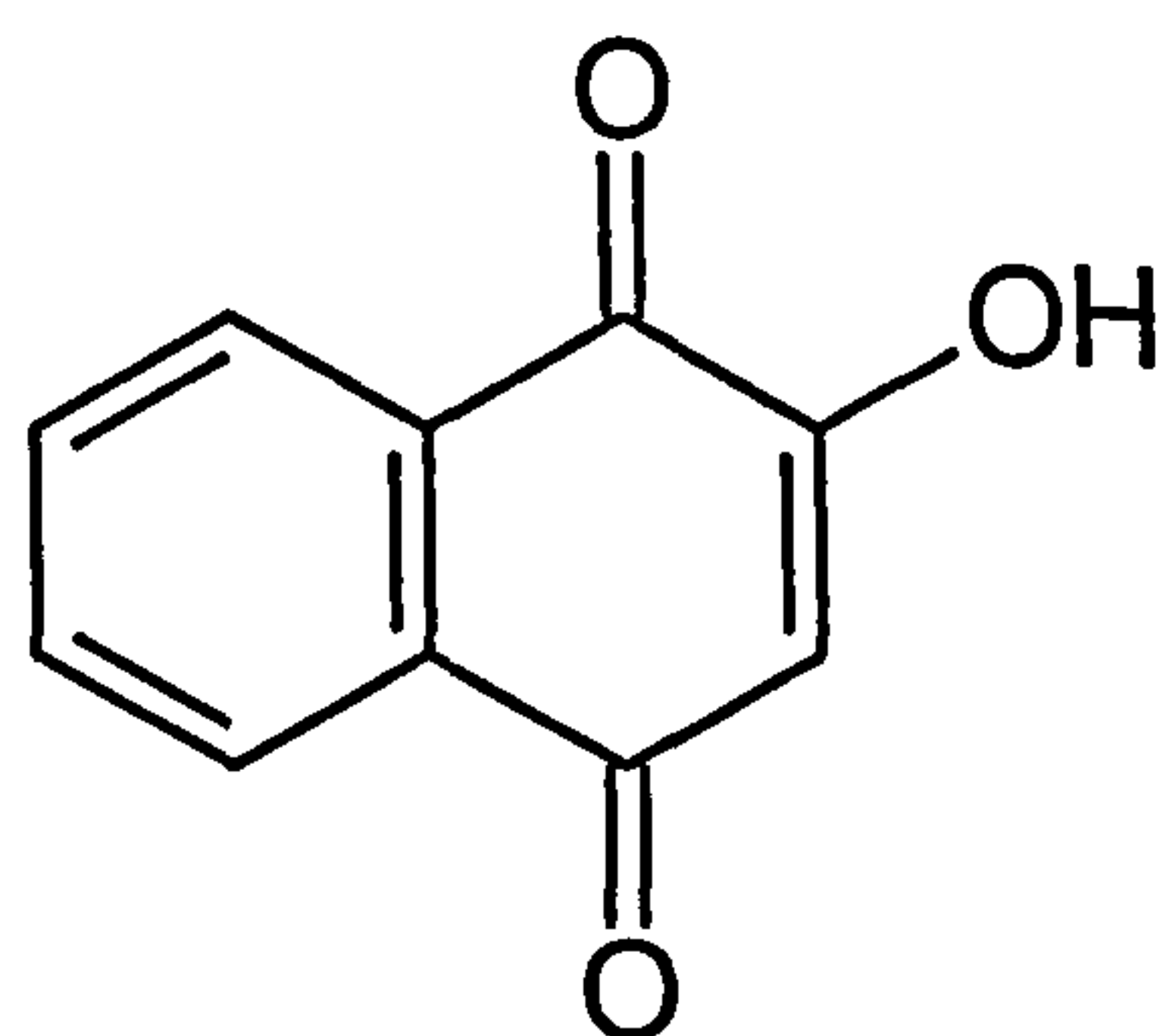
Vegetable dyes merit inclusion in studies of mummification, if only for their potential symbolic significance. Obtained from the native Egyptian shrub *Lawsonia inermis*, henna is an orange-red dye whose colour derives from the compound lawsone (2-hydroxy-1,4-naphthoquinone) (see Fig. 2.10) (Mills & White 1994, p.144). Whilst only a minor component, this could be a useful biomarker for henna and survives in an archaeological environment, as evidenced by the colour on both the hands (Lucas 1989, p.310) and hair (Fletcher 2000, p.500) of Egyptian mummies.

The petals of safflower (*Carthamus tinctorius*) contain two dyes, carthamin (red) (see Fig. 2.10) and an as yet unknown (yellow) (Mills & White 1994, p.144). Carthamin contains two six carbon rings and a sugar moiety, and likely to undergo oxidative degradation it can be difficult to predict the resulting degradation products. Although studies are lacking, possible products are 4-hydroxybenzoic acid and 2,3,4,6-tetrahydroxybenzoic acid, the latter sufficiently unusual and involatile to have potential as a biomarker.

2.3.11 Bitumen

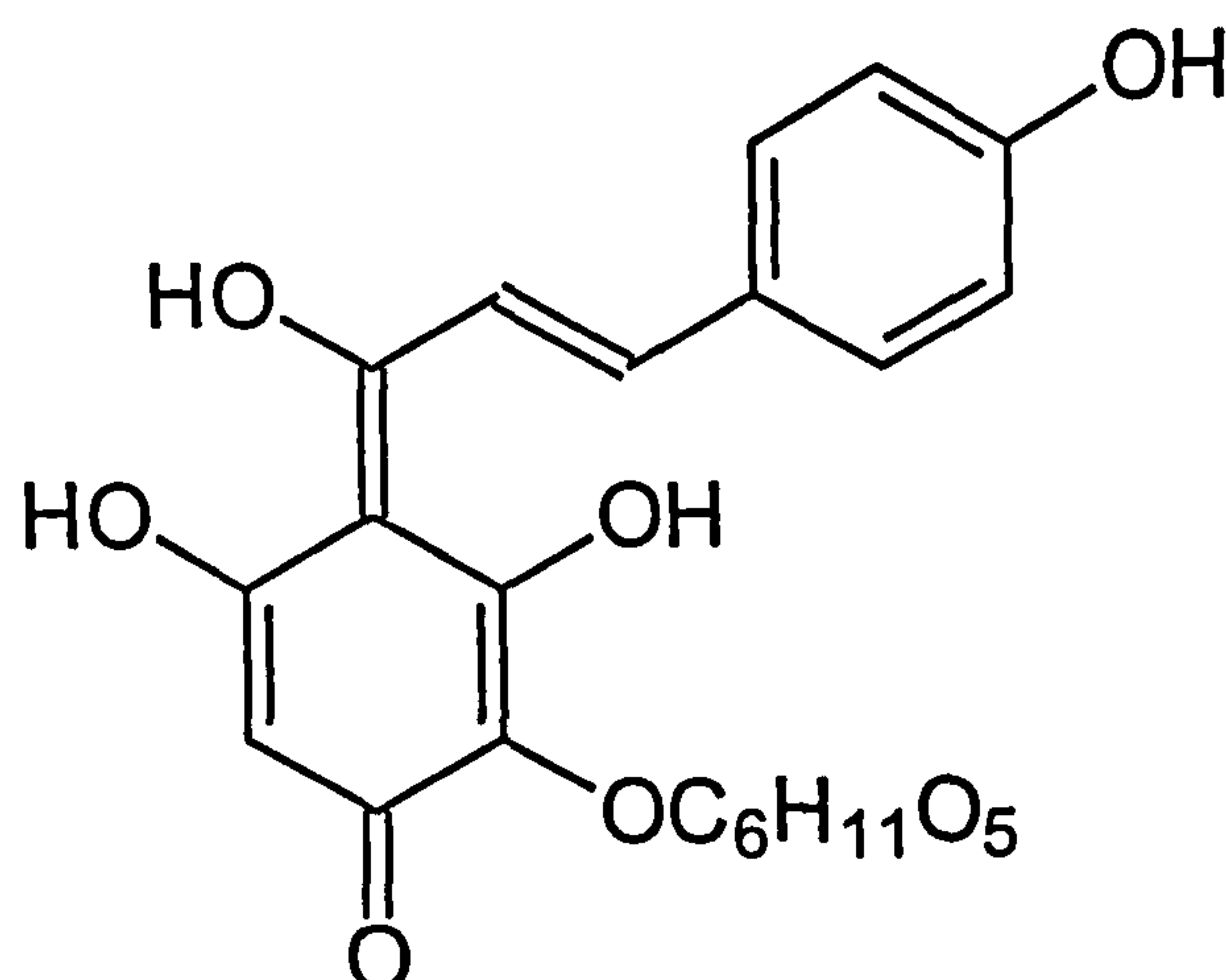
Natural bitumens (or 'asphalts') are petroleum products resulting from the loss of the more volatile components of crude oil. The remaining heavier fraction is natural 'bitumen', which can occur as either seepages from source rocks or from solid blocks such as those of the Dead Sea region (see Chapter 1). The chemistry of bitumens/asphalts has been extensively studied and consists of a hydrocarbon solvent-soluble fraction (the 'maltenes') and a polar, insoluble fraction (the 'asphaltenes') (Mills & White 1994, p.56-59). Of particular interest are the biomarker compounds of bitumen which derive from the components present in the original organisms from which the bitumens have been formed. Although they have undergone defunctionalisation and saturation/ dehydrogenation, they retain the carbon skeleton of the particular organisms' molecular input, allowing these biomarkers to be used to identify particular bitumens using a fingerprint approach (McKirdy et al. 1994, p.265-286; Gallegos 1971, p.1151-1160; Kimble et al. 1974, p.173-198; Richardson & Miller 1982, p.765-768).

Using this approach, Dead Sea bitumen has been found to lack diasteranes and exhibit a relatively high abundance of gammacerane and low pristane/phytane ratio of 0.5

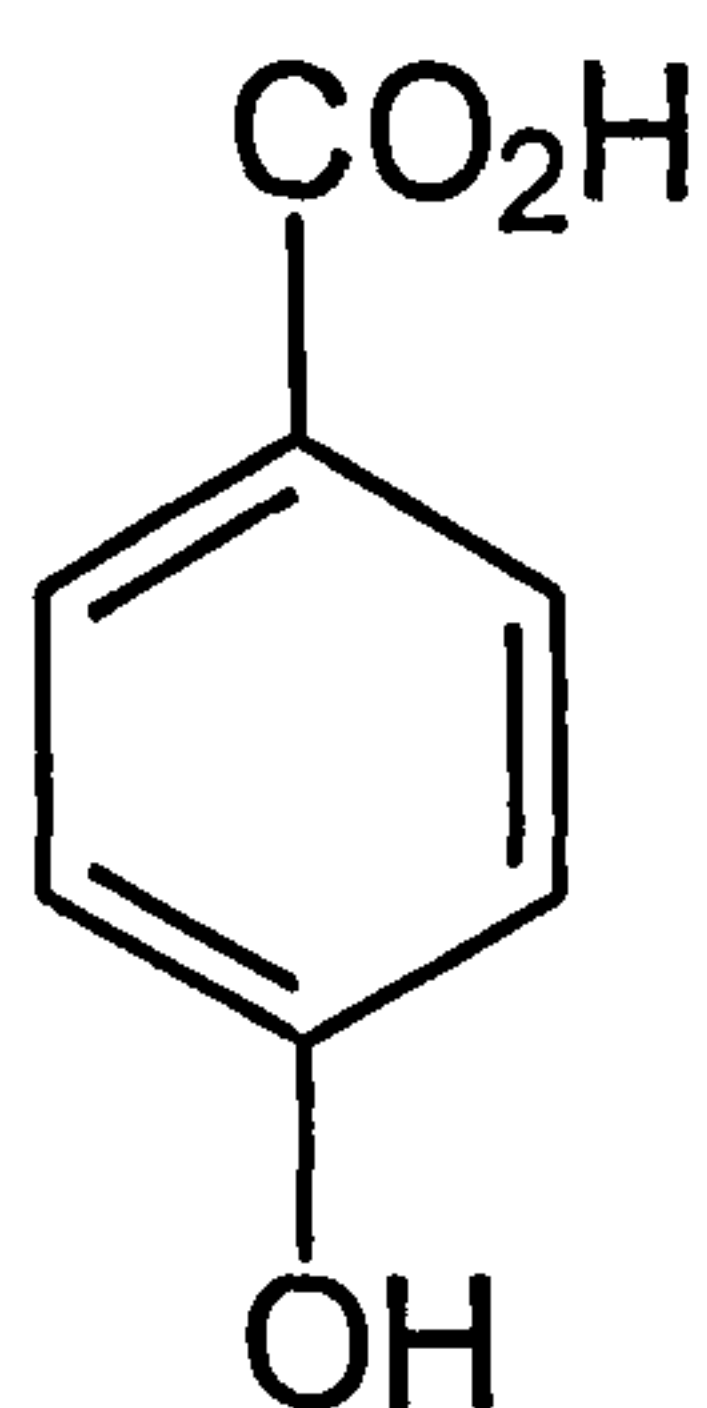


lawsone

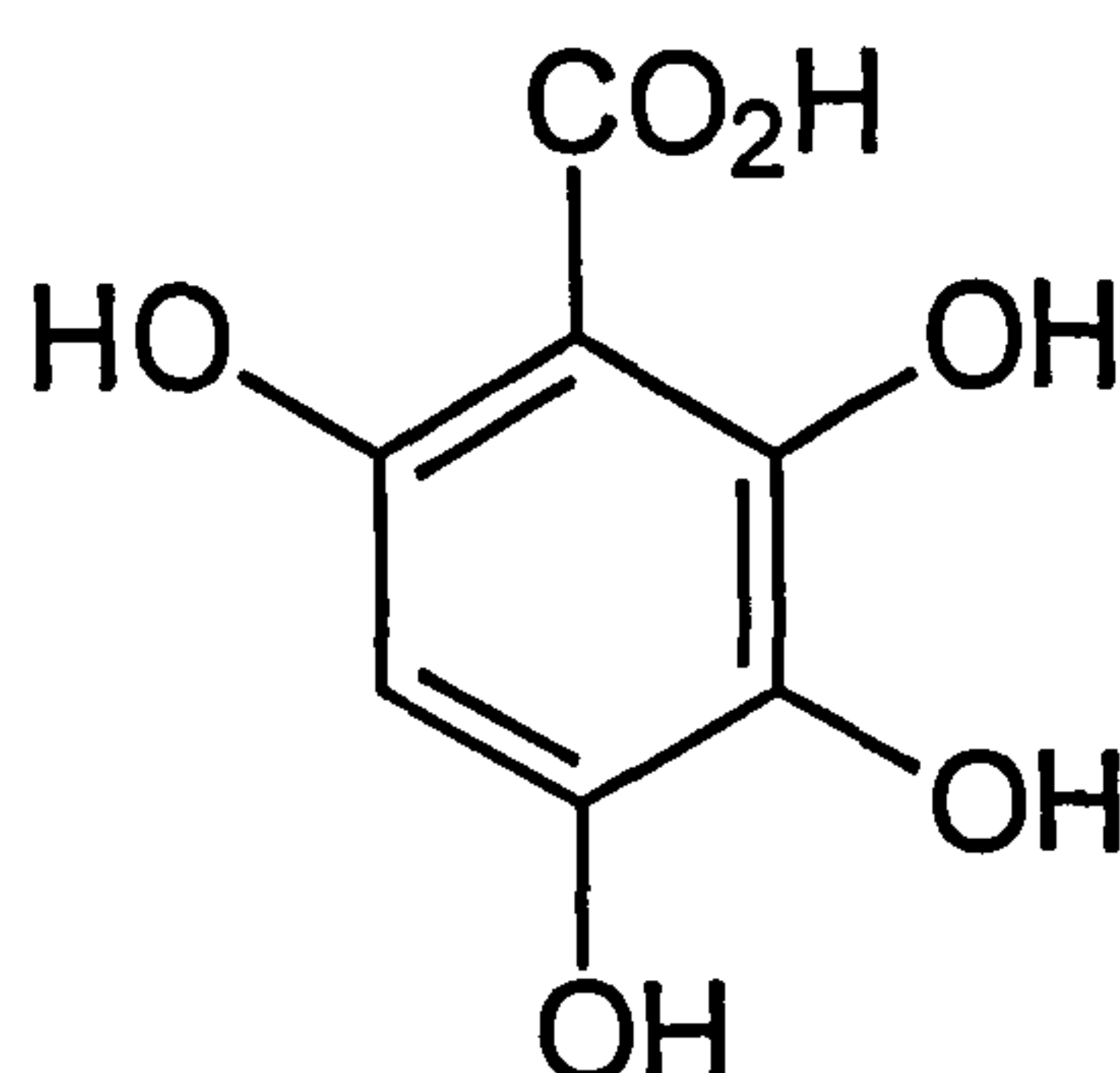
(2-hydroxy-1,4-naphthoquinone)



carthamin



4-hydroxybenzoic acid



2,3,4,6-tetrahydroxybenzoic acid

Figure 2.10 The structures of dye components occurring in vegetable dyes. Although they do not occur in the fresh dyes, 4-hydroxybenzoic acid and 2,3,4,6-tetrahydroxybenzoic acid are possible degradation products which could be expected to be observed in archaeological samples if safflower were present.

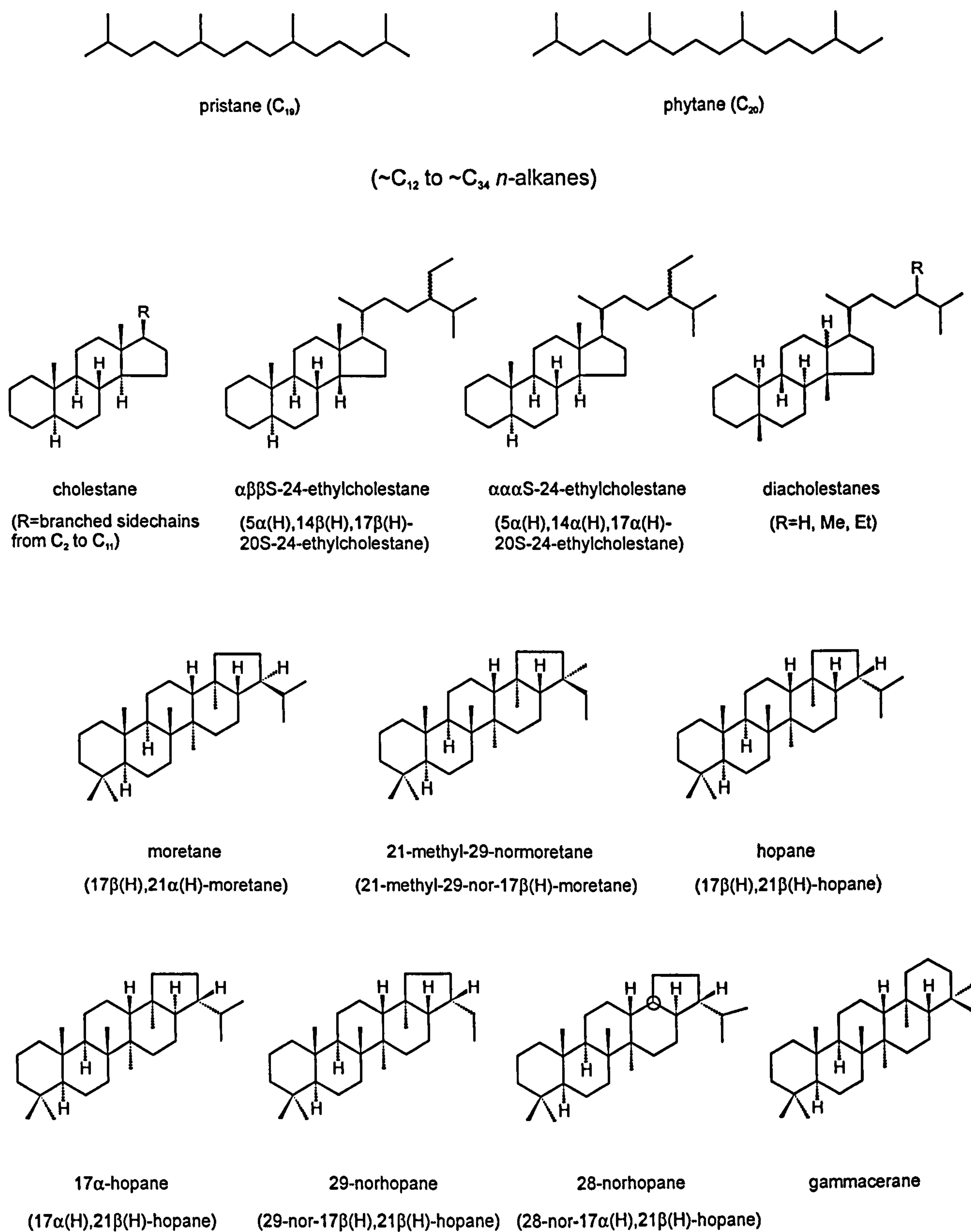


Figure 2.11 The structures of alkane, sterane, moretane, hopane and gammacerane components occurring in natural bitumens.

(Rullkötter & Nissenbaum 1988, p.618-621; Nissenbaum 1992, p.1-6; Connan et al. 1992, p.2743-2759). The $C_{15}+$ alkanes (with no odd-over-even-predominance), pristane and phytane, steranes, moretanes and hopanes (see Table 2.1 and Fig. 2.11) are biomarkers which can be used to identify bitumens and their geographical source (Rullkötter & Nissenbaum 1988, p.618-621; Nissenbaum 1992, p.1-6; Connan et al. 1992, p.2743-2759; Connan 1999, p.33-50). The hopanes are the most abundant of these characteristic biomarkers, their structures having received detailed study (Ourisson et al. 1979, p.709-729). The most important of these are 28-norhopane, 29-norhopane and hopane, evidence for which has been found in a number of archaeological samples (Mills & White 1994, p.192; Colombini et al. 2000, 19-29).

2.3.12 Honey

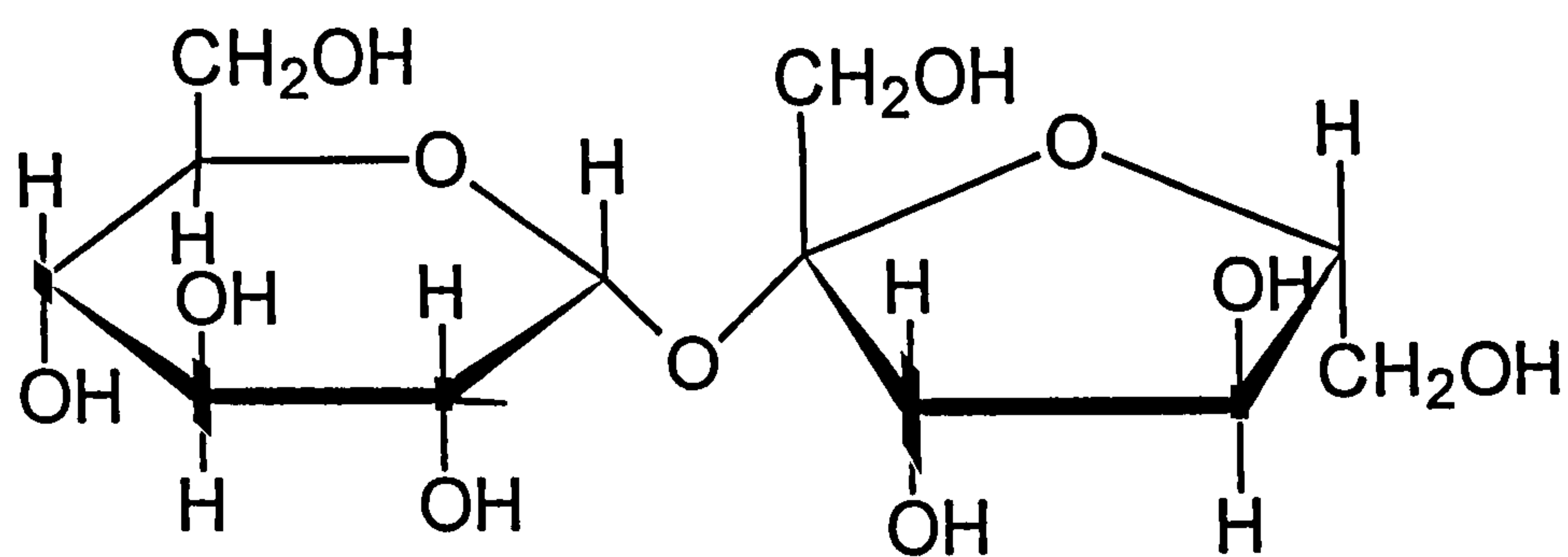
Honey comprises largely of the disaccharide sucrose with small amounts of free glucose and fructose (see Fig. 2.12) (Mills & White 1994, p.72), large quantities of which could suggest a sugar origin.

2.3.13 'Lotus' flowers

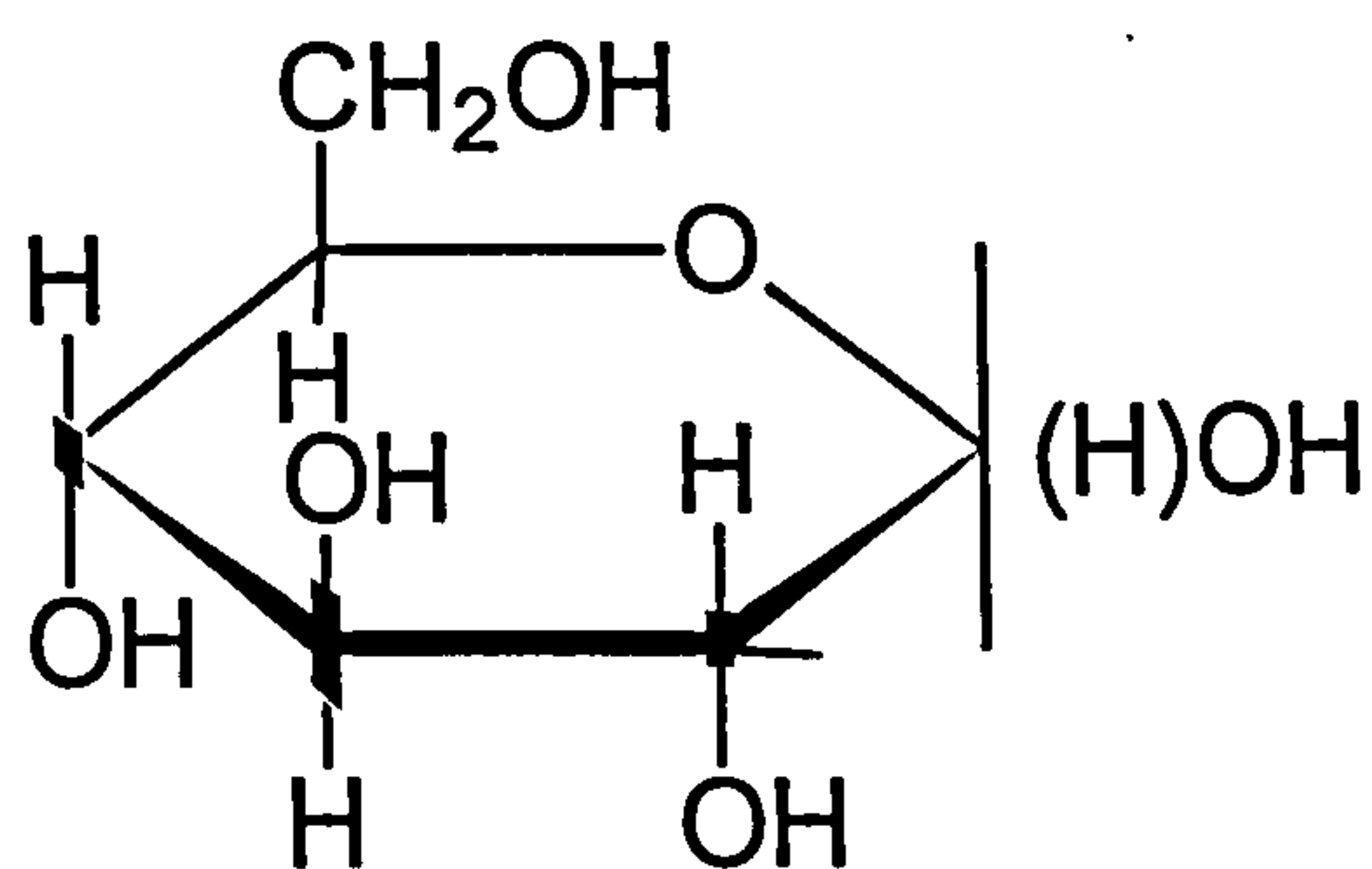
The blue lotus (*Nymphaea coerulea*) contains anthocyanidin galloylgalactosides (see Fig. 2.13) (Fossen & Andersen 1999, p.1185-1188; Fossen et al. 1999, p.1133-1137), the hydrolysed anthocyanidin components of which should persist in archaeological samples.

2.3.14 Date palm

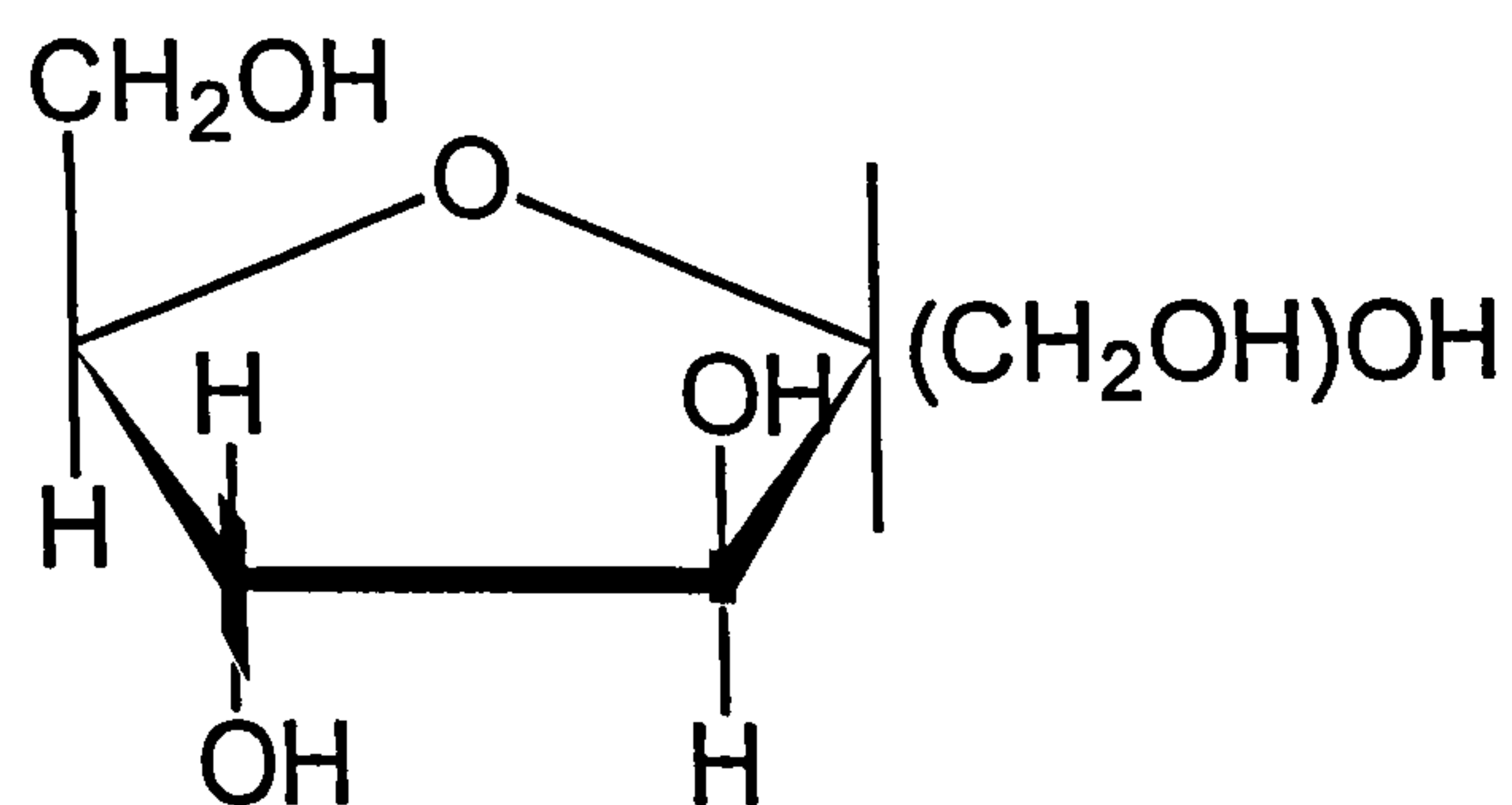
The date palm (*Phoenix dactylifera*) was used by the ancient Egyptians to produce wine, although whether this was by fermentation of the tree sap rather than the dates is unclear (see Fig.1.5.14). Although the fatty acids would be minor components of the wine of whichever source, they may still be present. Palm oil from the trunk contains a high abundance of palmitic acid and only smaller abundances of the other saturated acids ($C_{14:0}$ and $C_{18:0}$) likely to survive (Gunstone et al. 1986, p.98-99; McCance & Widdowson 1998, p.79) whereas oil obtained from the fruit would contain lauric acid as the major fatty acid with lower but significant abundances of myristic acid and a relatively low abundance of palmitic acid (Gunstone et al. 1986, p.98-99). Clearly, there would be difficulties in identifying such a commodity, given the volatility of the majority of the components, but



sucrose



D-glucose



D-fructose

Figure 2.12 The structures of polysaccharide and monosaccharide components occurring in honey. There are two possible anomeric forms (α and β) for each of the monosaccharides.

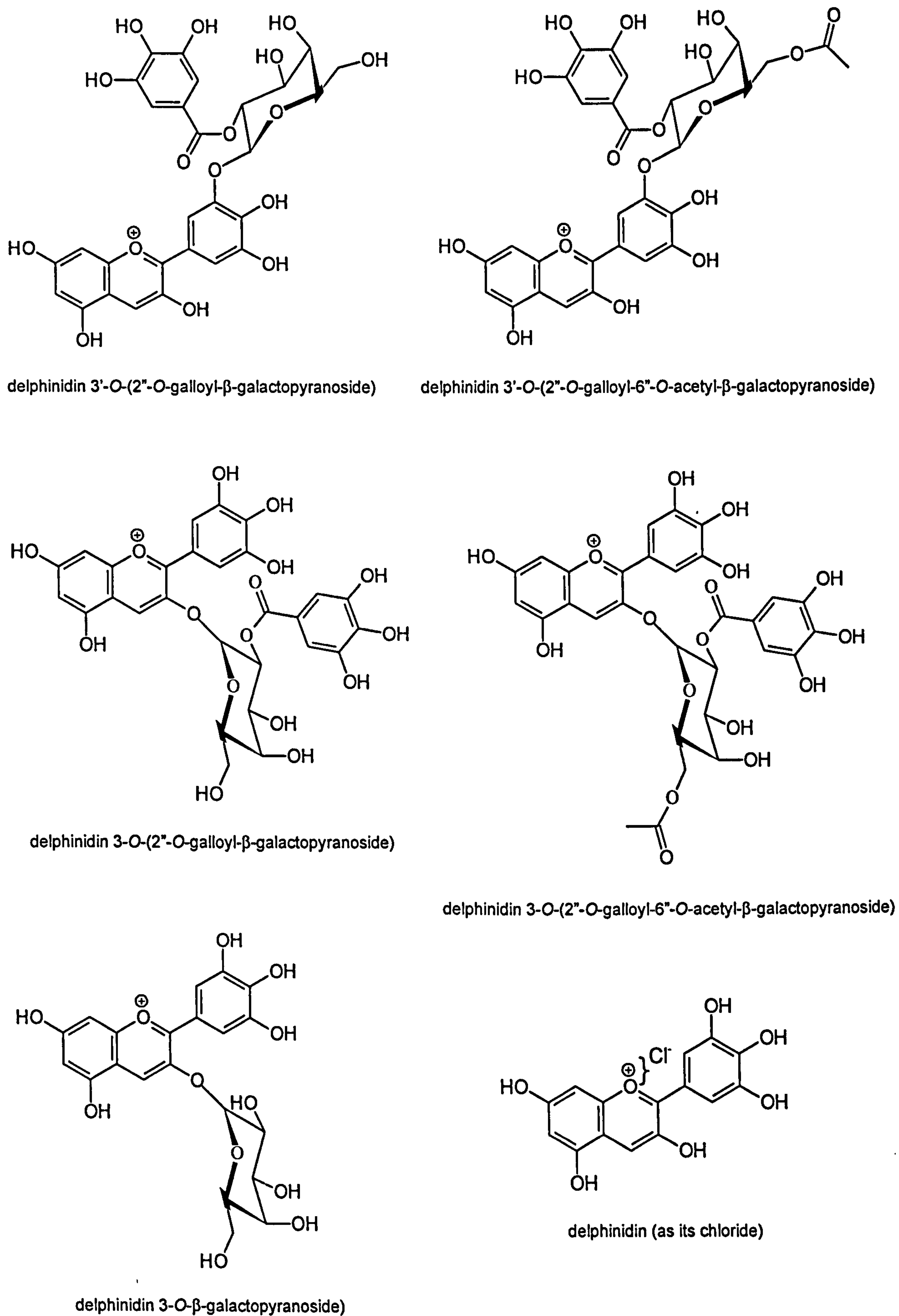


Figure 2.13 The structures of anthocyanins (anthocyanidin galloylgalactosides) and free anthocyanidin (as its chloride) occurring in the flowers of the blue lotus (*Nymphaea coerulea*).

its importance is too great to omit.

2.3.15 Sawdust

Sawdust consists of the biopolymer lignocellulose (Mills & White 1994, p.80; Faix *et al* 1991, p.213-219; Galletti & Bocchini 1995, p.815-826), the building blocks of which can be similar to both sugars and certain balsamic resins. If lignin is present, the guaiacyl and syringyl units (see Fig. 2.14) which are the building blocks of the polymer should also be identifiable (see analytical approach), confirming a lignin based product (i.e. sawdust) rather than a balsamic resin, which would otherwise contain similar aromatic acids (vanillic, protocatechuic and syringic acids) to those resulting from degraded lignin (Susic & Alongi 1997, p.243-253).

2.3.16 Storax

Storax resin from the tree *Liquidamber orientalis* is dominated by cinnamic esters with aromatic and triterpenoid acids in appreciable abundance (Mills & White 1994, p.109; Pastorova 1998, p.1381-1393) (see Table 2.1 and Fig. 2.15). The major components are cinnamyl cinnamate and 3-phenylpropanyl cinnamate, with significant amounts of benzoic and cinnamic acids, and the alcohols 3-phenylpropanol and cinnamyl alcohol. The triterpenoids present are oleanonic and 3-epi-oleanolic acid (Huneck 1963, p.479-482; Pastorova 1998, p.1381-1393) and these, together with significant quantities of cinnamic acids, could be used to identify storax resin in archaeological samples.

Styrax officinalis, has also been suggested as the source of the ancient "storax (or styrax) resin" (Hanbury 1857, p.465), although the modern plant produces no resin. Its chemical composition is said to be similar to gum benzoin (*Styrax* sp.) (Lucas 1989, p.95), which contains coniferyl benzoate as the major component (~75% of the gum; Schroeder 1968, p.57-61), with lesser amounts of p-coumaryl benzoate (Schroeder 1968, p.57-61). The coniferyl moiety is susceptible to oxidation, vanillic acid being produced along with free benzoic acid (present in the fresh gum), and with essentially no cinnamic acids and characteristic triterpenoid acids it would be possible to differentiate it from 'true' storax.

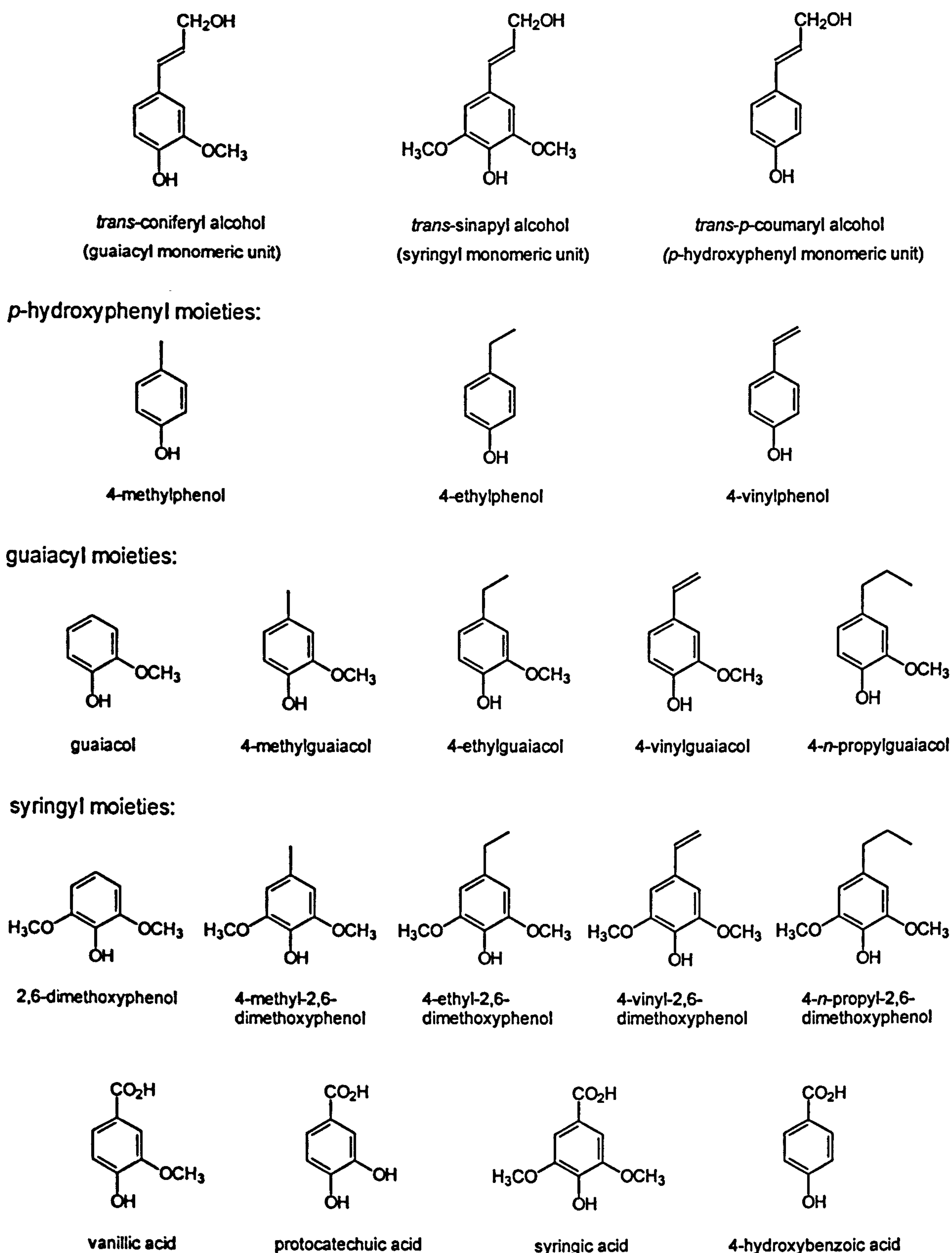
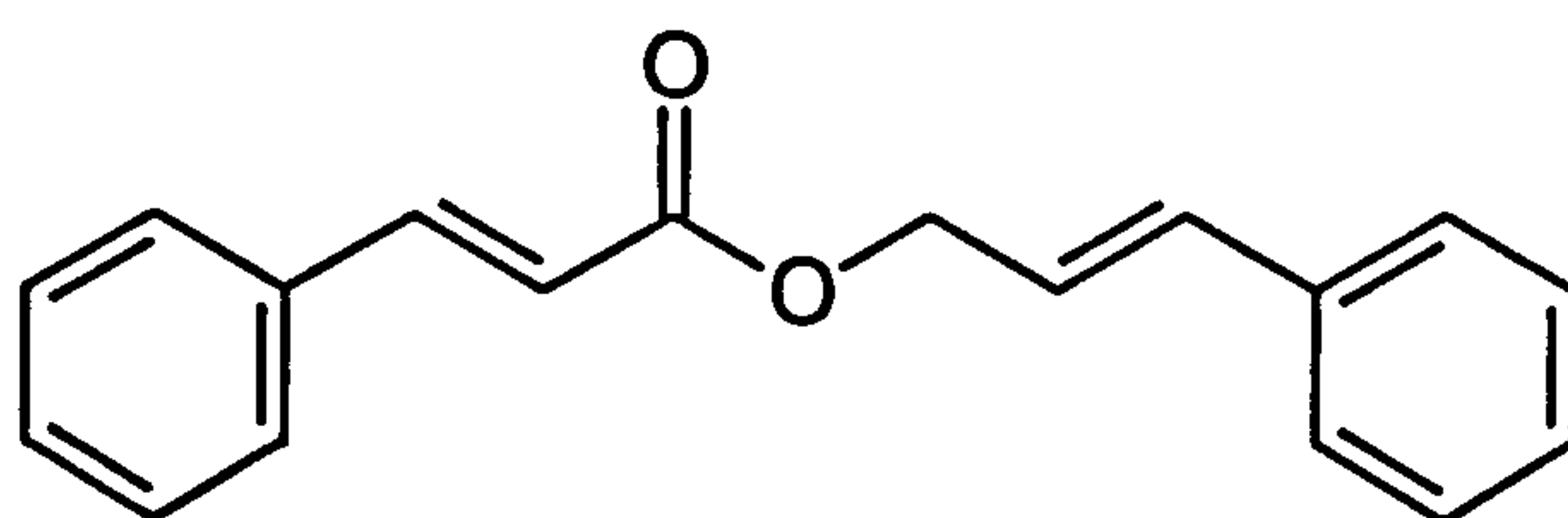
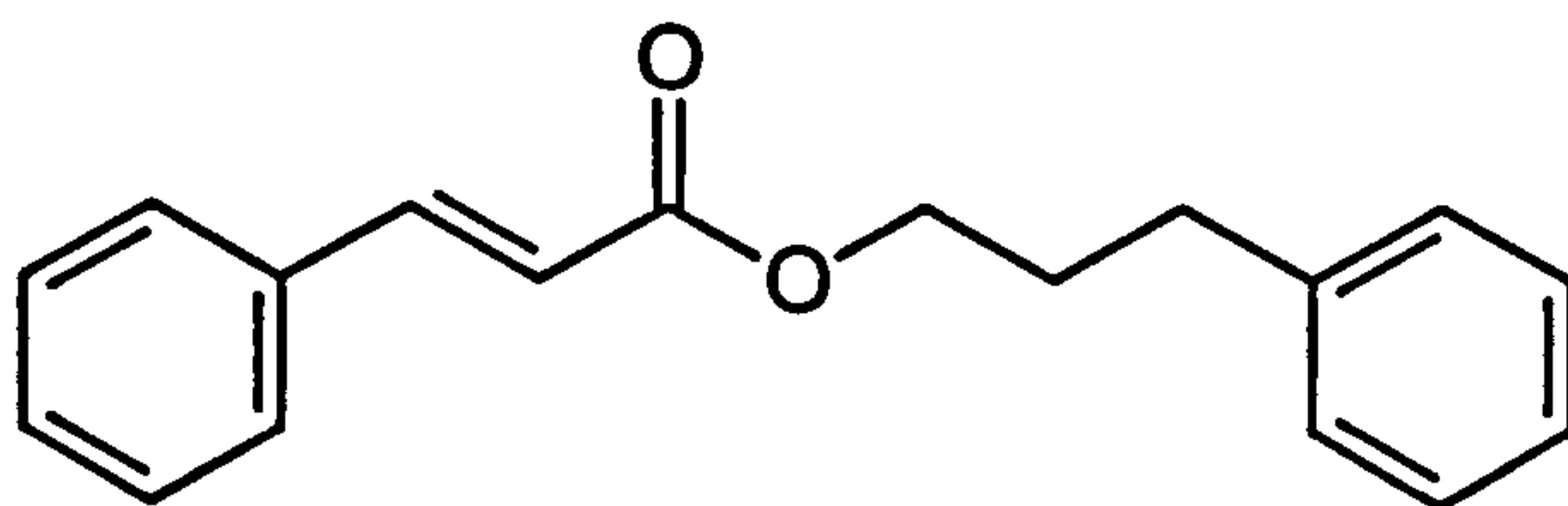


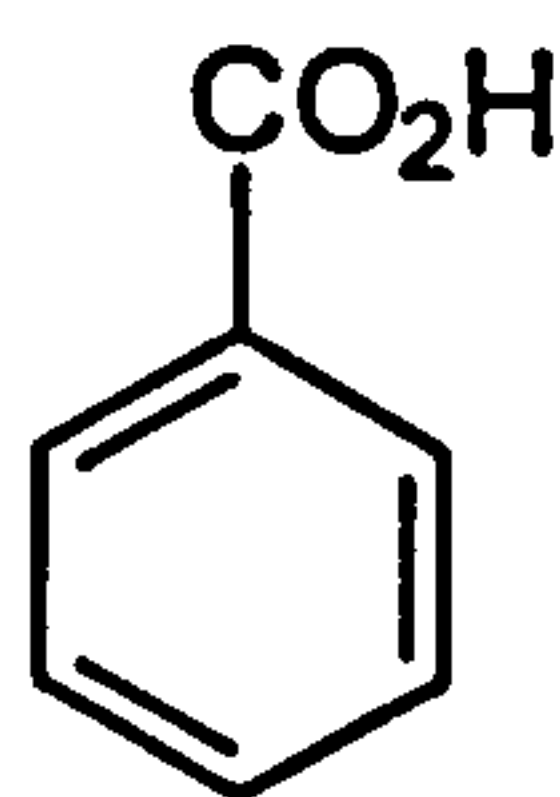
Figure 2.14 The structures of lignin components occurring in sawdust (wood). Coniferyl, sinapyl and coumaryl alcohol are the precursor building blocks which make up the lignin polymer. The analytical pyrolysis products of lignin are *p*-hydroxyphenyl, and the more diagnostic guaiacyl and syringyl moieties characteristic of lignin. Although they do not occur in freshly cut wood, vanillic, protocatechuic, syringic and hydroxybenzoic acids are oxidation products of degraded lignin and could be expected to be significant components in aged archaeological samples of a wood (sawdust) origin.



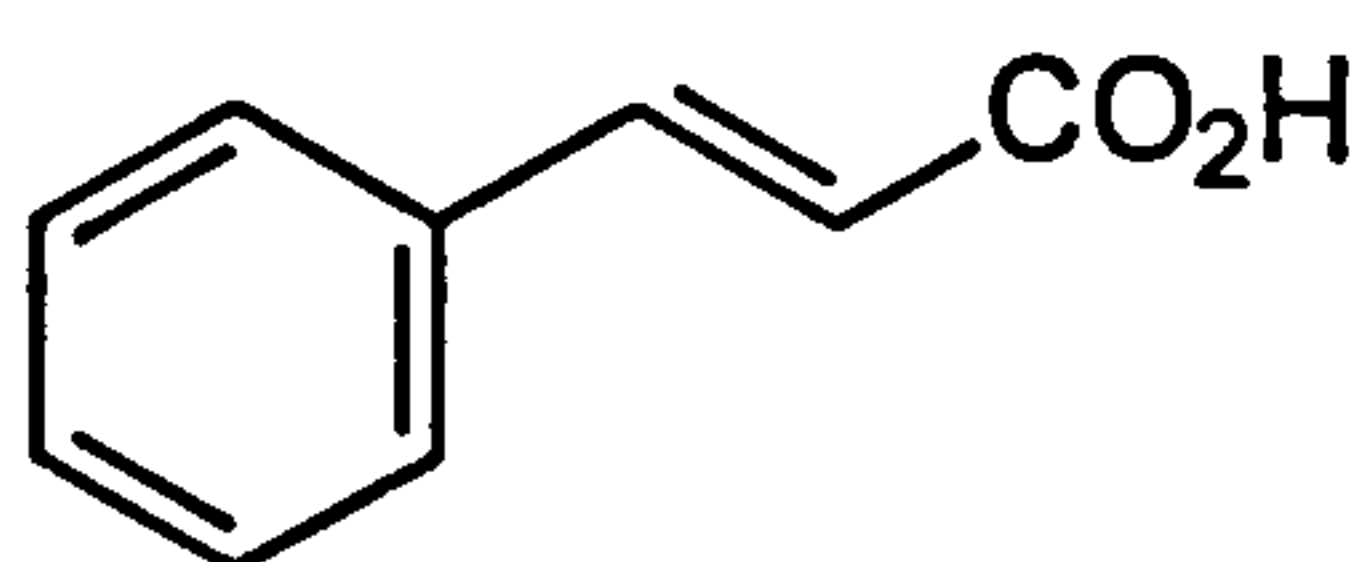
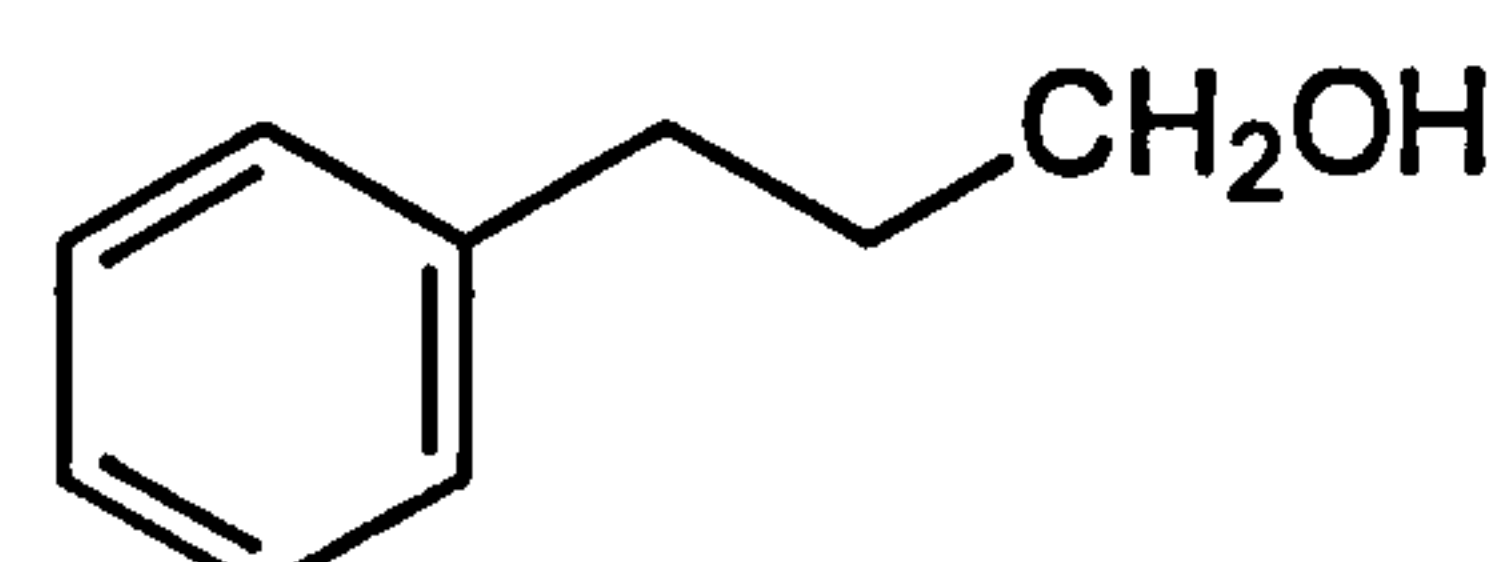
cinnamyl cinnamate



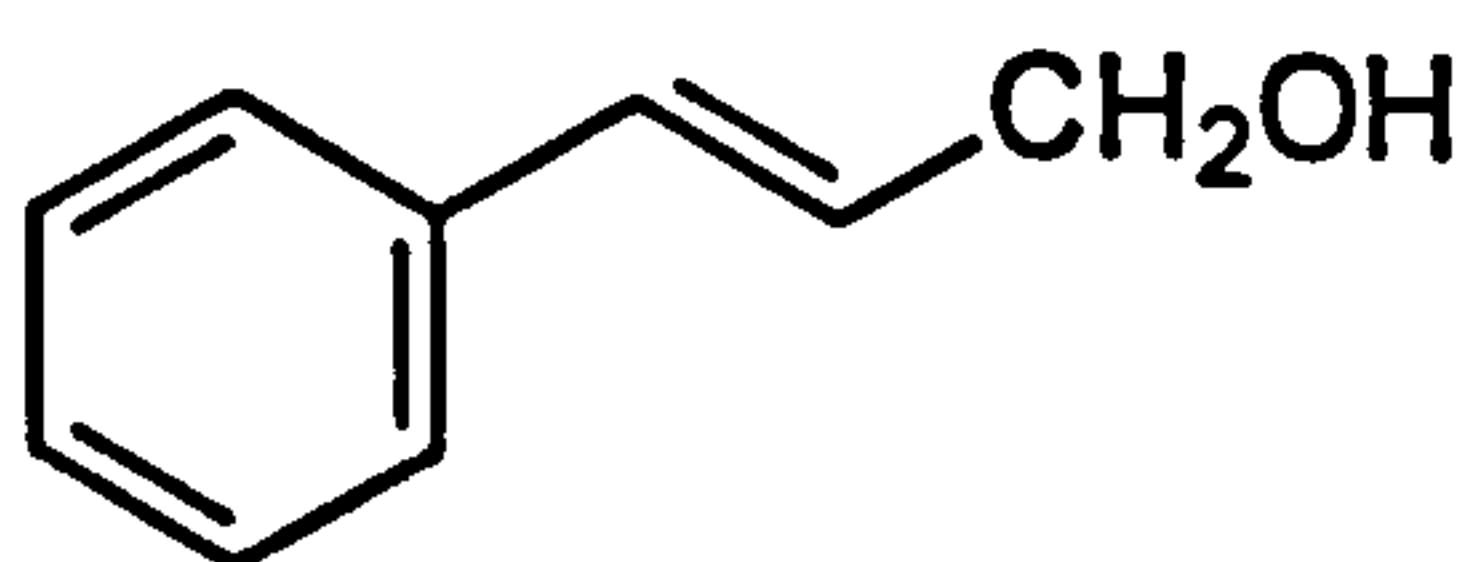
3-phenylpropyl cinnamate



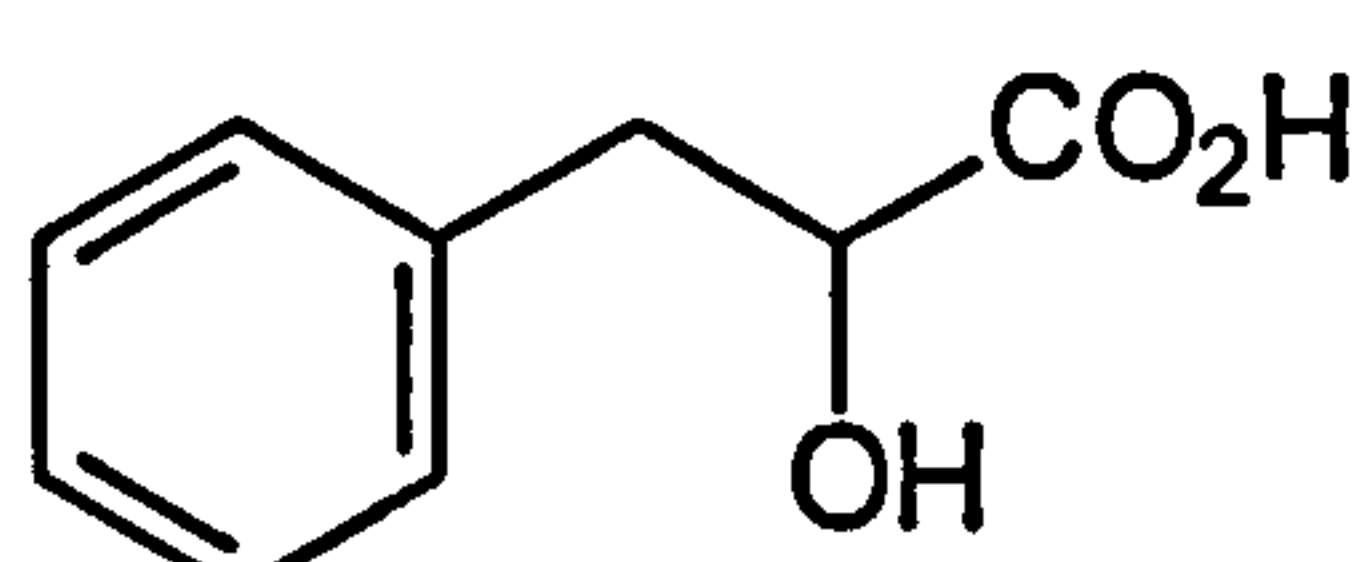
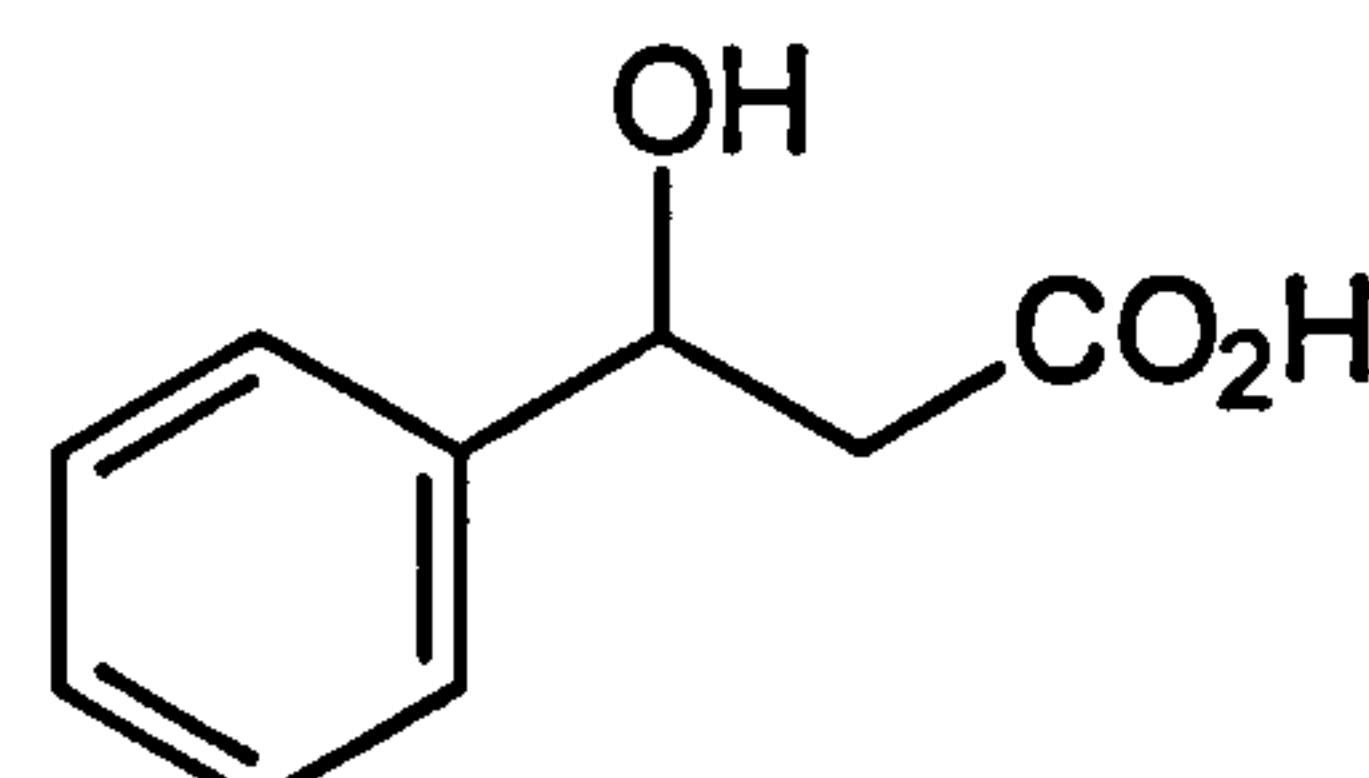
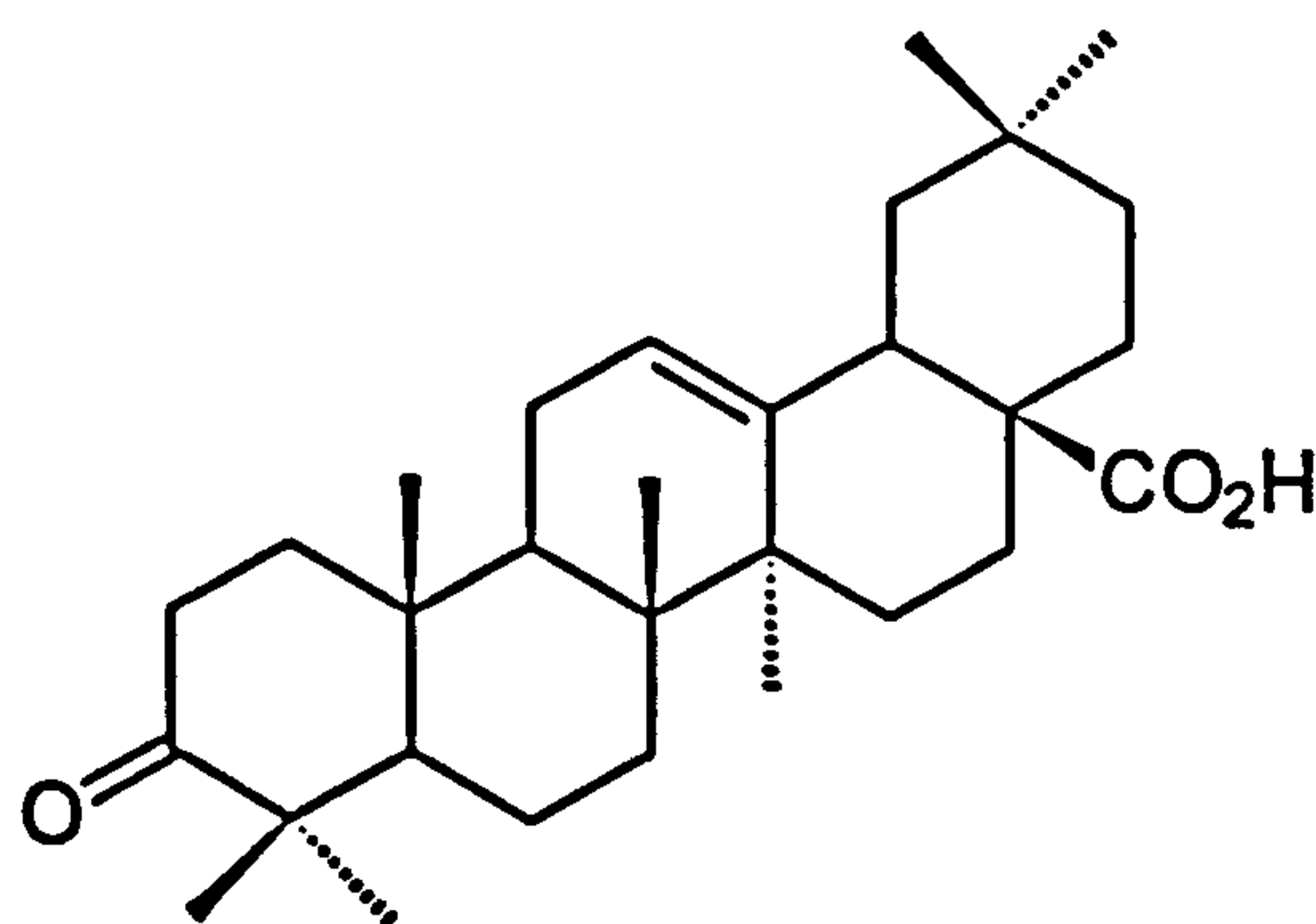
benzoic acid

*trans*-cinnamic acid

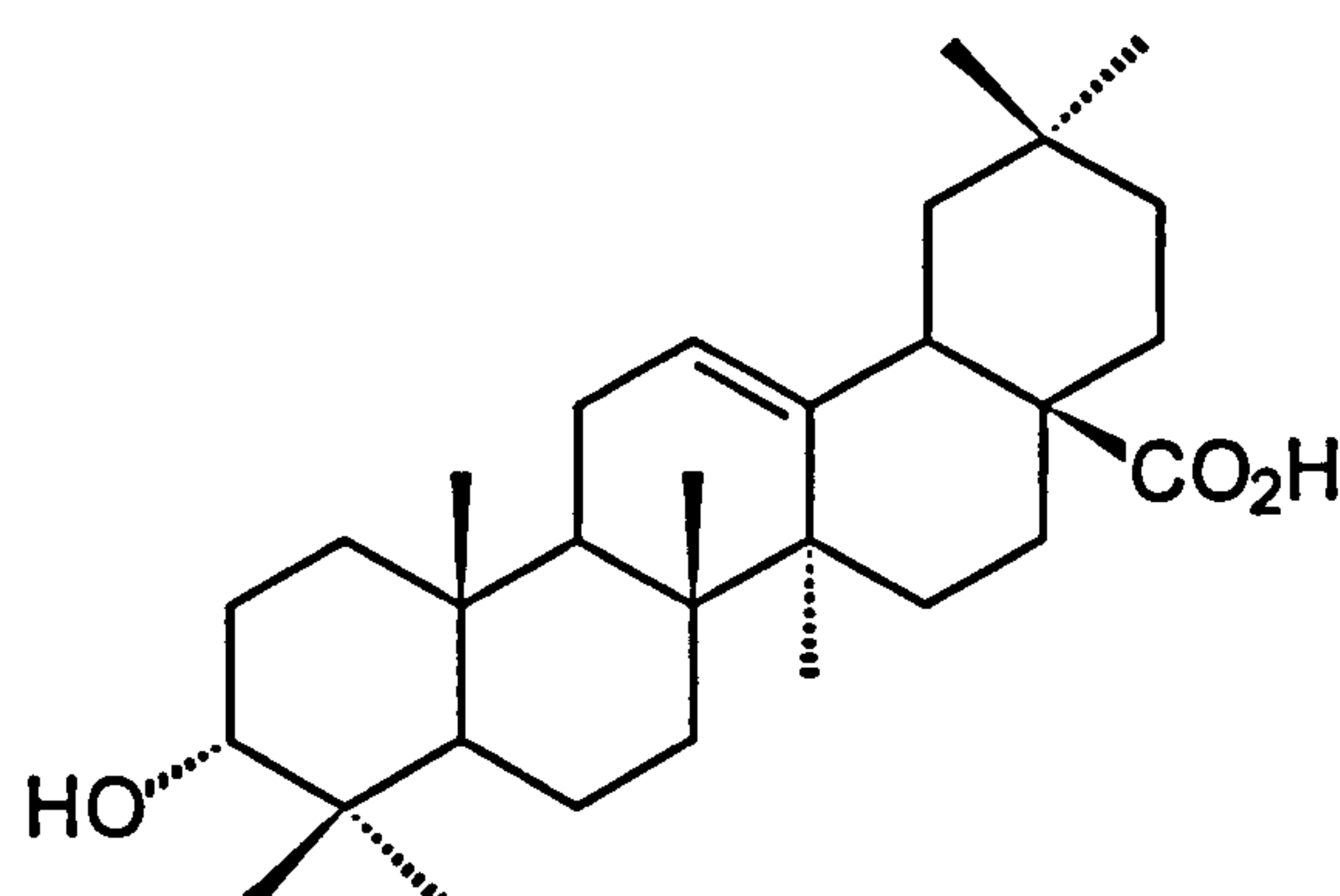
3-phenylpropanol



cinnamyl alcohol

 α -hydroxyhydrocinnamic acid β -hydroxyhydrocinnamic acid

oleanonic acid



3-epi-oleanonic acid

Figure 2.15 The structures of aromatic and triterpenoid components occurring in storax (*liquidamber orientalis*). Although they do not occur in the fresh resin, the α - and β -hydroxyhydrocinnamic acids are possible degradation products which could be expected to be observed in archaeological samples if storax was present.

2.3.17 Summary

It should be recognised that there are obvious difficulties when attempting to identify complex organic mixtures, which may have been admixed with other such substances and are often likely to have been subject to thermal treatments. The chemistry of the organic materials involved is generally complex, with the major compounds present in these aged degraded mixtures often absent from the literature. Consequently the significance of the biomarkers found can be uncertain, particularly given their presence in a range of aged materials, all of which could equally well have been utilised. Yet with extreme care it should be possible to elucidate the nature and origin of many these amorphous organic mixtures, thereby providing clues to the understanding of not only mummification, but other interrelated facets of ancient Egyptian culture.

2.4 PREVIOUS CHEMICAL INVESTIGATIONS INTO THE NATURE AND ORIGIN OF THE ORGANIC EMBALMING MATERIALS EMPLOYED IN ANCIENT EGYPTIAN MUMMIES

Surprisingly few of the large numbers of Egyptian mummies to have survived have been the subject of rigorous analytical studies. Knowledge of the precise nature of the materials used remains seriously lacking, with the vast majority of discussions relating to mummification based on nothing more than casual observation, supposition and a vivid imagination. Even the results of studies undertaken in recent years are inaccurate in their assumption that the subject is now fully understood (Bahn 1992; Connan 1988, 1991, 1999, etc.). Table 2.2 presents a chronological summary of the chemical analyses which have been carried out in order to determine the nature of the organic materials employed in embalming.

The earliest recorded chemical examination of an Egyptian mummy was undertaken by G.F. Rouelle as early as 1750. Described as “*very systematic work*”, it nevertheless consisted of “*distilling the samples and resulted in no useful or certain data*”, the only conclusions drawn being that “*the distillates obtained resembled the products of the distillation of amber*” (Lucas 1908, p.141). Among more general ‘unwrappings’ of Egyptian mummies during the early 19th century (e.g. Granville 1825, 269-316; Pettigrew 1834), that carried out in 1828 by Leeds Philosophical Society involved the examination of the mummy of the priest Natsefamun who lived at Karnak in Upper Egypt during the reign

TABLE 2.2. Previous chemical investigations of the organic embalming materials in ancient Egyptian mummies

AUTHOR	MUSEUM	MUMMY DATE	PROVENANCE	SEX/AGE/NAME	SAMPLE ORIGIN	TECHNIQUES	FINDINGS
Lucas 1908	Cairo	1390-1352 BC	Thebes	Male adult ('Amenhotep III')	Cheeks, left arm, pelvis	Solubility, Acid/ester/ saponification values, 'Spot tests'	Gum resin: myrrh Gum resin: myrrh Not identified
				Male adult ('Ramses IV')	Cranium, mouth		Gum resin: myrrh Gum resin: myrrh
				Male adult ('Siptah')	Face		Gum resin: myrrh
				Male young adult	Cranial cavity		Not identified
				Female adult ('Tawosret')	3 rd left rib, soles of feet		No resinous material
				Female adult ('Nesikhonsu')	Unspecified		Not identified
				Female adult ('Priestess of Amen')	Cranial cavity (x4), scalp, face		Resin: not yet identified Myrrh Myrrh
Lucas 1911	Cairo	See Lucas 1908	See Lucas 1908	See Lucas 1908	See Lucas 1908	Solubility Fluorescence	Resins Bitumen absent
Reutter 1912	Neuchatel	380-343 BC Unspecified Unspecified Unspecified Unspecified	Unspecified Unspecified Unspecified Unspecified Unspecified	Unspecified	Unspecified	Sulphur content 'Spot tests' smell	Bitumen
				Unspecified	Unspecified		Bitumen
				Unspecified	Unspecified		Bitumen
				Ibis	Unspecified		Bitumen
				Bird bandages	Bandages (from various birds)		Bitumen
Spielmann 1932	Cairo (from Lucas)	1069-945 BC 664-525 BC	Unspecified Unspecified	Unspecified	Canopic box	UV fluorescence, Spectrographic:	Bitumen
				'Resin from ? royal mummy' 'Resin'			Materials 'occupy positions between the undoubted

Table 2.2. contd.			332-30 BC Unspecified	Unspecified Unspecified	Unspecified Unspecified	Cranial/thoracic packing, skin 'A mummy'	Ni, Mo, V	bitumens and the undoubted resins'
Griffiths 1937	Cairo (from Lucas)		Unspecified	Unspecified	Unspecified	'?Wood pitch, from mummy'	Solubility, Saponification value, 'Spot tests' Spectrographic: Ni, Mo, V	Coniferous pitch
Zaki & Iskander 1943	Cairo		c.525-404 BC	Sakkara	Male adult (‘Amentefnekt’)	Packing material, material from above body, material from underneath body	Solubility, Saponification value, Sulphur content Ni, Mo, V spectrographic, UV spectrographic	Bitumen
Farag & Iskander 1971	Cairo		c.1800 BC	Hawara	Female adult (‘Neferuptah’)	Fragment from waterlogged mummy debris inside sarcophagus	Solubility, Combustion	Resin & ferric oxide
Coughlin 1977	Philadelphia Art		c.300-30 BC	Unknown	Male adult (‘PUM II’)	Unspecified	XRD/TLC	Essential oils of juniper, <i>cinnamomum camphora</i> , myrrh
Benson et al. 1979	Manchester		c.AD 380	Unknown	Female young adult	Outer 4 layers of wrappings	TLC/GC/MS AAS, Lassaigne test	Beeswax, galbanum, bitumen (on innermost wrappings)
Storch & Schäfer 1985	Munich		Unspecified	Unknown	Unknown	1.Outer coating 2.Wrappings 3.Body surface 4.Thorax and skull	IR/XRF/IC/MS	1.Beeswax, oil 2.Beeswax, oil, tree resin, bitumen, gum, soda 3.Beeswax, oil 4.Beeswax, oil, bitumen, gum, soda, fossilised resin(?)
Wright & Wheals 1987	Various		2000 BC-AD 395	Unspecified	Various	Various cartonnages	Py/MS	Polysaccharide gums, rosins, waxes
Goyon & Josset 1988	Lyon Guimet		c.150 BC-AD 90	Unknown	Male adult	Linen between knees (x2) Visceral packing ‘balm’ Skull ‘balm’	GC/MS	Beeswax, gum resins, bitumen

Table 2.2. contd. Rullkötter & Nissenbaum 1988	London British	c.900 BC c.200 BC c.100-AD 200 c.100-AD 200	Thebes Akhmim Thebes Thebes	Male(?) adult (‘Pasenhor’) Male(?) adult (‘Djedoler’?) Unspecified Unspecified	Coffin Mummy Wooden mummy board Wooden mummy board	GC/MS	Bitumen, plant waxes
Connan & Dessort 1989	Lyon Guimet	c.150 BC-90 AD	Unknown	Male adult	Linen between knees (x2) Visceral packing ‘balm’ Skull ‘balm’	GC/MS	Beeswax, gum resins, bitumen
Connan & Dessort 1991	Cairo	c.1250 BC	Unknown	Unspecified	Unspecified	GC/MS	Bitumen (Dead Sea), conifer, beeswax
	Kestner, Hanover	c.600-500 BC	Unknown	Unspecified	‘Resin’ from wrappings around knees		Beeswax, bitumen (Iraq)
	Kestner	c.200 BC	Unknown	Unspecified	‘Resin’ from wrappings around feet		Bitumen (Iraq)
	Musee de l’Homme	c.300-50 BC	Unknown	Unspecified	‘Resin’ from near bone of right iliac		Bitumen (Dead Sea)
	In situ	c.0-AD 300	Queens’ Valley, Thebes	Unspecified	Abdominal cavity		Beeswax, conifer, Bitumen (Dead Sea)
	In situ	c.0-300 AD	Queens’ Valley, Thebes	Unspecified	Abdominal cavity		Bitumen (Iraq)
Connan 1991	Lyon Guimet	c.300 BC	Unknown	Unspecified	Foot	GC/MS	Bitumen (Iraq), conifer, beeswax
Nissenbaum 1992	Review of previous work	See Rullkötter & Nissenbaum 1988; Connan & Dessort 1989	See Rullkötter & Nissenbaum 1988; Connan & Dessort 1989	See Rullkötter & Nissenbaum 1988; Connan & Dessort 1989	See Rullkötter & Nissenbaum 1988; Connan & Dessort 1989	GC/MS & HPLC	Bitumen, beeswax, conifer, gum

Table 2.2. contd. Proefke et al. 1992	Illinois Urbana- Champaign	c.100-AD 200	Unknown	Child(?)	Wrappings, wooden board, crystallised 'resins' from feet	GC/MS	Conifer resin, bitumen (although no hopanes!)
Kaup et al. 1994	Staatliche Antiken- sammlung	c.300-200 BC	Unknown	Unspecified	Unspecified	GC/MS	Pistacia, oil of cedar?, oil of turpentine?
Taylor 1995	Bristol	c.1040 BC	Thebes	Male adult (<i>'Horemkenesi'</i>)	Back & spine	GC/MS	Plant waxes, tree resins
Mejanelle et al. 1996	Lyon Guimet	c.100 BC	Unknown	Male adult	Unspecified	GC/MS	Beeswax?, resin, fat/oil
	Lyon Guimet	Unspecified	Unknown	Crocodile	Unspecified		Beeswax, fat/oil, resin?
	Venice	c.300 BC	Unknown	Unspecified	Unspecified		Fat/oil, gum
Mejanelle et al. 1997	Lyon Guimet	c.100 BC	Unknown	Male adult	13 areas of body inc. right side of pelvis, right knee, right toes, package of internal organs "containing heart" [sic!]	GC/MS	Vegetable tannins
Fletcher & Rogers 1997	in situ	3400 BC	Hierakonpolis	Female adult	Cranial hair	TLC/AS	Tannin based colorant
	in situ	3400 BC	Hierakonpolis	Male adult	Cranial hair		Tannin based colorant
	Qasr el-Einy	2000 BC	Deir el-Bahari, Thebes	Female adult (<i>'Ashayet'</i>)	Cranial hair		Tannin based colorant
	Qasr el-Einy	1460 BC	Deir el-Bahari, Thebes	Male adult (<i>'Harmose'</i>)	Cranial hair		Yellow colorant, gum
Koller et al. 1998	Hildesheim	c.2150 BC	Giza	Male adult (<i>'Idu'</i>)	Clavicle fragments	GC/MS	Coniferous resin/tar
Serpico & White 1998	Boston Fine Arts	c.2000-1750 BC	Deir el-Bersha	Male adult (<i>'Djehutinakht'</i>)	Canopic jar 'black resinous deposit'	GC/MS	Pinaceae (not fir)
	London British	c.700 BC	Unknown	Female adult	1.Chest cavity 2.Back of skull		1.Cedar or pine pitch (strongly heated) 2.Pistacia resin pitch
Weser et al. 1998	Hildesheim	c.2150 BC	Giza	Male adult (<i>'Idu'</i>)	Clavicle fragments	GC/MS	Coniferous resin/tar (heating/'smoking')

Table 2.2. contd. Connan 1999	Unspecified	1100-800 BC 750-400 BC 750-400 BC 750-400 BC 750-400 BC	Unknown Unknown Unknown Unknown Unknown	Unspecified Unspecified Unspecified Unspecified Unspecified	Unspecified Unspecified Unspecified Unspecified Unspecified	Unspecified Unspecified Unspecified Unspecified Unspecified	GC/MS	Bitumen, beeswax, fat?, conifer resin? Bitumen, beeswax, fat?, conifer resin? Conifer resin Fat?, conifer resin? Bitumen, beeswax, conifer resin Bitumen, beeswax, fat?, conifer resin Bitumen, beeswax, conifer resin Bitumen, beeswax, fat?, conifer resin? Fat?, conifer resin Bitumen, fat? Conifer resin?	Bitumen, beeswax, fat?, conifer resin? Bitumen, beeswax, fat?, conifer resin? Conifer resin Fat?, conifer resin? Bitumen, beeswax, conifer resin Bitumen, beeswax, fat?, conifer resin? Fat?, conifer resin Bitumen, fat? Conifer resin?
	Manchester Bristol	1900 BC 1050 BC	Rifeh Deir el-Bahari, Thebes	Male adult (‘Khnumnakht’) Male adult (‘Horemkenesi’) Female adult (‘Merneith’)	Bandage/resin/tissue Resin-like material	Unspecified Unspecified Unspecified Unspecified Unspecified	GC/MS, Sequential TD- GC/MS, Py- GC/MS	Fat/oil, proteinaceous material Fat/oil	Fat?, conifer resin? Fat?, conifer resin Bitumen, fat?, conifer resin?
	in situ	c.600 BC	Sakkara	Portion of resinous mass from left side of thorax	Unspecified	Unspecified	GC/MS	Mastic resin (Pistacia), unidentified vegetable oil, beeswax, bitumen	Mastic resin (Pistacia), unidentified vegetable oil, beeswax, bitumen
	London British	c.700 BC	Unknown	1.Chest cavity 2.Back of skull	Unspecified	Unspecified	GC/MS	1.Coniferous pitch, beeswax, 2.Conifer pitch, Pistacia resin, beeswax	1.Coniferous pitch, beeswax, 2.Conifer pitch, Pistacia resin, beeswax

of Ramesses XI (1099-1069BC). The first interdisciplinary investigation of an Egyptian mummy to be undertaken, the chemical analysis reported the presence of natron, together with myrrh, cassia, gelatine, tannins and 'resin'. The team of investigators also noted that Natsefamun's mummy had "*a thick layer of 'spicery' covering every part of it which still retains the faint smell of cinnamon...but when mixed with alcohol or water and exposed to heat the odour of myrrh becomes very powerfully predominant*" (Osburn 1828). Yet given the limited nature of the forms of testing available at that time, namely solubility in various solvents, simple chemical 'spot' tests and often little more than their own sense of smell, their findings cannot be regarded as entirely conclusive. The only other significant investigation of the 19th century was carried out in 1888 by E.M. Holmes, and included a sample of so-called 'resin' from the wrappings of a mummy of the Roman Period (c. AD 2nd– 4th century). On heating in a flame, the material gave off what was described as the irritating "*vapours of benzoic acid*" and it had a "*decided vanilla odour*" (Holmes 1888, p.387-389). The identification of the sample as benzoin based on the odours produced on heating is clearly unsatisfactory, as is the conclusion for the other 'resin' sample investigated, which was identified as Chian turpentine based on its solubility in alcohol, its odour, and the texture and taste when chewed in the mouth!

The first serious investigation into the organic preservatives used in mummification was undertaken by A. Lucas in 1908, involving analysis of samples derived from several New Kingdom royal mummies (see above) (Lucas 1908, p.133-147). His investigations were based on a series of simple chemical tests involving solubility in various solvents, the saponification, acid and ester values, and simple 'spot' tests (e.g. colour reactions), with odour also used to identify the materials present. His identification of myrrh (Lucas 1908, p.143) was based upon the acid and saponification values for myrrh, but because these values can change substantially over time (see Griffiths 1937, p.703-709) as degradation takes place, any attempt to use the values of a fresh resin to identify samples taken from mummies several thousand years old was necessarily flawed.

A coniferous resin, "*possibly cedar*" (Lucas 1908, p.143), was identified in one sample as a result of Liebermann-Storch reaction in which a purple colour 'indicates' a coniferous resin. Yet given the wide range of resinous materials available, and the inherent difficulties with interferences in colour reactions such as this, the conclusion cannot be considered safe. The physical appearance of samples was also then considered a useful indication of a 'resin' being present, yet a material which is widely considered to be a resin simply on

account of its form (e.g. Taylor 1985, p.92) can often be nothing of the sort (Buckley & Evershed, forthcoming). Lucas did in fact admit the difficulties in identifying the organic materials, particularly the resins, using the chemical tests then available. Yet in 1911 Lucas published the results of a thorough chemical investigation which included the aforementioned work carried out three years earlier, and which is rightly regarded as a rigorous scientific piece of work which can still be referred back to for useful information on embalming materials (Lucas 1911). Based on solubility and fluorescence of the samples in solution, he concluded that bitumen was not employed in Pharaonic mummies (c.3100-332BC).

In 1912 L. Reutter analysed material from six mummies, only one of which was dated, and this to the very end of the dynastic period (XXXth dynasty, human). Bitumen was identified in all six samples on the basis that the blackish residue separated out contained sulphur, the residue of one sample reduced sulphuric acid to sulphurous acid upon heating, and that a smell "*characteristic of bitumen*" was noticed in one sample. Given that carbon disulphide which could contain free sulphur was used to dissolve the residue, the significance is questionable (Lucas 1989, p.305). Nor is the reduction of sulphuric acid to sulphurous acid at all specific for bitumen, with many organic materials producing the same result, and furthermore, the smell of aged, often highly degraded materials is clearly not a conclusive method of analysis.

In 1932 P.E. Spielmann looked at a number of mummy samples supplied by Lucas. Employing fluorescence and spectroscopic analysis of the samples' ash, the absence of any fluorescence was used to support the presence of bitumen, despite contradictory information given elsewhere in the same report (Spielman 1932, Table 2, p.179), with the resins considered the alternative sample origin giving fluorescence between yellow and red/brown (Spielmann 1932, p.178). It should be noted that the absence of fluorescence does not necessarily suggest bitumen, which in fact does fluoresce at certain wavelengths in an appropriate solvent (Zaki & Iskander 1943, p.240-241). The inconsistency in the presence and relative proportions of the three metals characteristic of bitumen, i.e. Nickel (Ni), Vanadium (V) and Molybdenum (Mo) determined spectroscopically, make the results from this study inconclusive so far as the use of bitumen is concerned.

In 1937 J.G.A. Griffiths examined four samples of 'resins' and 'pitch' from ancient Egyptian tombs (Griffiths 1937, p.703-709). Included in this study was "? wood pitch,

from mummy” and, like the other three samples was described as “a black lump”. Solubilities were the main criterion used to identify the materials as resins, wood pitch or bitumens. Saponification and acid values were also obtained, although Griffiths did concede that these values could change on aging as oxidative changes took place. Spectroscopy was also employed to determine the quantities of Ni, V and Mo as an indicator for bitumen, although none were found present in the ‘black lump’. Chemical ‘spot’ tests were also employed to identify the ‘resins’, including the Liebermann-Storch reaction and the abietic acid test which produce characteristic colours for certain substances. The Liebermann-Storch reaction produces a strong purple colour for cedar resin (Griffiths 1937, p.705), whereas in the abietic acid test cedar, sandarac and mastic produce a blue green colour and myrrh gives a negative reaction. Yet any such colour changes are susceptible to interference, and given the likely degraded nature of the materials being analysed together with the likelihood that more than one substance may be present, these tests can be only of very limited value.

In 1943 A. Zaki and Z. Iskander used a variety of techniques to identify resin/bitumen mixtures in three samples obtained from the mummified body of Amentefnekht (XXVIIth dynasty, 525-404BC) (Zaki & Iskander 1943, p.223-255). Based on solubilities, it was suggested that the resin in question, present in all three samples, could be either a gum or true resin, whereas more notably it was concluded that the samples also contained appreciable amounts of bitumen based on solubility, sulphur content, UV spectroscopy, and spectrographic analysis for the characteristic bitumen elements Ni, V and Mo. These are the first results to strongly suggest that bitumen may indeed have been employed in embalming, although the lack of specificity of the tests employed means that the results cannot be regarded as conclusive.

In 1971 Z. Iskander collaborated with S.A. Saleh in the chemical analysis of samples from the Hawara burial of Neferuptah, daughter of Amenemhat III (1855-1808 BC). These included a sample of “black organic matter” believed to have been used in the mummification process, with its appearance, behaviour on ignition and solubility in alcohol used to conclude that the sample contained a resin with the addition of ferric oxide (Farag & Iskander 1971, p.121-122), a conclusion which clearly cannot be regarded with any degree of certainty.

Only two years later in 1973, the first interdisciplinary study of the modern age took place in Detroit, Michigan when the Pennsylvania Mummy Team led by A. Cockburn examined an anonymous male mummy of Ptolemaic date ('PUM II'). Chemical analysis was carried out on the so-called 'mummy fluids', with X-ray diffraction used to identify oil of the genus *juniperus*, and the minor components from *Cinnamomum camphora* and myrrh from *Commiphora myrrha* identified by TLC (Coughlin 1977, p.7-8). Yet the basis for these conclusions is not made clear. It is difficult to reconcile such a precise, species-specific origin for the minor components using TLC, a technique which in itself lacks specificity, and although the identification of the genus *juniperus* is interesting, its significance is difficult to assess in the absence of any actual chemical data in the report.

As part of the Manchester Museum Mummy Project in 1979, wrappings from an unprovenanced female mummy c. AD 380 were examined using amino acid analysis (Benson et al. 1979, p.119-131). The identification of gelatin challenges the general assumption that plant gum rather than animal glue was used to secure mummy wrappings (see Herodotus II.90, 1954, p.161), although given that the wrappings analysed date from the very end of the Roman period they cannot be taken as representative of ancient Egyptian practices.

The same study also identified bitumen, based on the presence of sulphur (Lassaigne sodium fusion test) together with vanadium (NAA) and molybdenum (AAS). Unfortunately the Lassaigne test is qualitative rather than quantitative, giving no information on the amount of sulphur present, which although unlikely, could even be inorganic. The absence of appreciable quantities of the nickel found with vanadium and molybdenum in bitumen makes doubtful the conclusion that bitumen was present in the sample analysed, as indeed is admitted in the study's conclusion (Benson et al. 1979, p.124).

However, GC/MS data obtained from the same sample indicates that beeswax is clearly present. Perhaps the most interesting finding however, is the identification of galbanum obtained from the area of Iran (ancient Persia) (Benson et al. 1979, p.126, 128). Nevertheless, the use of TLC to identify the characteristic compound umbelliferone from such an aged, complex mixture must remain open to question, and further work would be needed to confirm the discovery.

In 1985 Storch and Schafer analysed samples taken from the wrappings, head, body surface and body cavity of an undated, unprovenanced mummy of unspecified sex (Storch & Schafer 1985, p.327-338). A combination of ion chromatography, infra-red (IR), X-ray fluorescence and MS was used to identify the components present. The outer coating was identified as a mixture of beeswax and oil, with beeswax and oil also found on the wrappings together with tree resin, bitumen, gum and soda. The skin surface was found to be coated with a mixture of oil and beeswax, and material found in both the skull and thoracic cavity consisted of beeswax, oil, bitumen, gum, soda and fossilised resin. The nature of this fossilised resin is unclear, it would certainly be a point of interest which would merit further clarification.

Chemical analysis was also undertaken by Wright and Wheals in 1987 of samples from ancient Egyptian mummy cases using pyrolysis-mass spectrometry (Wright & Wheals 1987, p.195-211). General classification into gums, rosins or waxes was possible in only half of the samples, and a more precise origin was not possible due to the lack of a separation stage in the analysis. Separation of the components in these materials prior to MS could have produced a more precise molecular identification. Nevertheless, the study is valuable in terms of the number of samples analysed, albeit largely unprovenanced.

An unprovenanced Graeco-Roman mummy (c.150 BC-AD.90) was the subject of a study carried out by Connan and Dessort. Four samples were analysed, two taken from linen around the area of the knees, one from visceral packing and the other a 'balm' from the skull. GC/MS was used to identify the components present (Goyon & Josset 1988, p.103-107; Connan & Dessort 1989, p.1665-1672). Bitumen was found to be present in all four samples, evidenced by the presence of steranes, terpanes and phenanthrenes, with two distinct sources of Dead Sea bitumen apparent from differences in their sterane distributions, the bitumen associated with the viscera differing from the other three samples. The main components of the balms were a coniferous pitch indicated by dehydroabietic acid and the aromatised diterpenoid retene, beeswax confirmed by the presence of C_{23} - C_{35} *n*-alkanes and C_{40} - C_{48} wax esters, fatty acids indicating a fat or oil and possibly a gum resin characterised by cadalene (Connan & Dessort 1989, p.1669-1670, Fig.1, 4). However, cadalene can also be indicative of other resins such as dammar (van Aarssen 1992, p.63-88), and since it is a degradation product of gum resins its significance is therefore uncertain.

A Ptolemaic mummy from Akhmim (c.200 BC), in addition to two Roman mummy boards (c. AD 100-200) and a XXIInd dynasty coffin (c.900 BC) were analysed by Rullkötter and Nissenbaum using GC/MS (Rullkötter & Nissenbaum 1988, p.618-621). Steranes and triterpanes with a high relative abundance of gammacerane and an absence of diasteranes were identified in the mummy and two mummy boards, patterns which are characteristic of Dead Sea asphalts (Rullkötter & Nissenbaum 1988, p.619; Nissenbaum 1992, p.1-6). The coffin also contained bitumen markers, i.e. steranes and hopanes, although these were from another and as yet unidentified source. The study also mentions the presence of plant waxes, although the *n*-alkane distribution C₂₃-C₃₅ with C₂₇ the major hydrocarbon (Rullkötter & Nissenbaum 1988, p.619, Fig.1) would in fact indicate a beeswax origin. Confirmation of the presence of the wax esters (C₄₀-C₅₂) present in beeswax would have been needed to firmly conclude this, but its apparent omission from the study is noteworthy, and it was in fact corrected at a later date (Nissenbaum 1992, p.1-6). The fact that the study also fails to mention exactly which part of either the mummy or burial equipment the samples originated also detracts from its value.

Further analysis by Connan and Dessort in 1991 used GC/MS of samples from six mummies, two intrusive burials in the Theban necropolis' Valley of the Queens (tombs QV.33, QV.73) and four of unknown provenance (Connan & Dessort 1991, p.1445-1452). Two of the unprovenanced Ptolemaic mummies were found to contain bitumen from two different sources, one from the Dead Sea and one from the area of modern Iraq. Of the two provenanced mummies of Roman date, one (QV.33) contained hydrocarbons indicative of beeswax, and the hydrocarbons, steranes and hopanes characteristic of Dead Sea bitumen together with longifolene, a component present in conifers. The other Roman sample (QV.73) also contained bitumen, but again from the alternative source in Iraq. Samples from two unprovenanced mummies of the dynastic period were also examined, the 'resin' taken from around the knee area of a Saite mummy c.600-500 BC proving to consist of beeswax and Iraqi bitumen whilst the oldest of the samples taken from a XIXth dynasty (1295-1186 BC) contained hydrocarbons characteristic of both beeswax and bitumen from the Dead Sea area. Longifolene was also the major component of the hydrocarbon fraction, indicative of a conifer.

In further analysis of samples, Connan also identified bitumen from Hir-Abu Jir in Iraq from the foot of a Ptolemaic mummy, whereas samples from two canopic jars c.600 BC

yielded bitumen from the Dead Sea region. The coniferous component longifolene, and beeswax was also identified (Connan 1991, p.35).

In 1992 a team in Illinois took samples from the feet of a mummified child and using GC/MS revealed the presence of the diterpenoids dehydroabietic acid, 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid indicating a degraded/oxidised coniferous resin. Hydrocarbons in the C₁₉-C₃₃ (C₂₃ max) range, with no-odd-over-even-predominance was used to conclude the presence of bitumen, although no steranes or hopanes were reported (Proefke et al. 1992, p.105-110A).

A sample of skeletal muscle tissue from a Ptolemaic mummy was analysed by a German team using GC/MS, with the major component identified as a pistacia resin and the most abundant compound being noroleanone indicative of a strongly heated resin (Kaup et al. 1994, p.489-500). Monoterpenoids, including α -pinene, cymene and limonene were identified, their presence being attributed to oil of turpentine from the Aleppo pine (*Pinus halepensis*). In fact these monoterpenes occur in Pistacia itself and although volatile may have survived the heating process. The possible presence of "cedar oil" is also reported, as evidenced by the identification of the characteristic sesquiterpenes, although the identification of cedrene as one of these substituted C₁₅ azulenes suggests a *Juniperus* origin (often the source of 'cedar oil') rather than a *Cedrus* (true cedar) species.

The findings of the analysis of samples taken from the mummy of the XXIst dynasty priest Horemkenesi were published in 1995, with so-called 'resin' reported as being "extracted from tree resins and plant waxes" (Taylor 1995, p.92). Re-examination of the original data showed that the GC/MS analysis had in fact revealed none of the terpenoids indicative of either a true resin or gum resin, and this has recently been corrected in a forthcoming report (Buckley & Evershed, forthcoming).

In 1996 samples from the unprovenanced Ptolemaic remains of two humans and a crocodile were analysed by GC/MS (Mejanelle et al. 1996, p.M.16-M.20). Material from the crocodile mummy revealed fatty acids indicative of a fat/oil and hydrocarbons characteristic of beeswax. Monoterpenoids were also identified, although no higher terpenoids indicative of the resinous component were detected. A sample from the earliest of the human mummies (c.300 BC) was found to contain fatty acids indicative of a fat or oil origin, with analysis of wrappings from the same mummy revealing both fatty acids and

carbohydrates indicating a fat/oil and gum mixture. A sample from the second human mummy (c.100 BC) revealed fatty acids, hydrocarbons and terpenoids indicative of a fat/oil, beeswax and a resin respectively. The same team later detected inositols and gallic acid (identified as their TMS derivatives) which confirmed the presence of vegetable tannins in the 'balms' and 'tissues' taken from this second human mummy (Mejanelle et al. 1997, p.177-186). They also found hydrocinnamic acid, vanillic acid and protocatechuic acid as their TMS derivatives, suggesting cinnamon or a balsamic resin such as styrax or benzoin. Although their results are of great interest, their emphasis on the polar components indicative of tannins creates something of an unbalanced picture.

A report in the same year by Fletcher and Rogers also identified tannins of a vegetable origin in the cranial hair of several mummified bodies (Fletcher & Rogers 1997). Most notable was the presence of tannins in a male and female mummy from the predynastic cemetery of Hierakonpolis, (c. 3400 BC), the earliest evidence for the application of an organic treatment to mummified remains (Fletcher 2000, p.500). The hair from the mummy of Queen Ashayet (c.2000 BC) also contained a tannin-based colorant, whilst that of the adult male Harmose (c.1460 BC) from Deir el-Bahari had been treated with a yellow colorant with the addition of what was described as 'a gum'. Further work on the precise nature of this yellow dye would be useful.

The earliest evidence to date for the use of a coniferous product in Egyptian embalming was reported by a German team who studied the Old Kingdom mummy of the adult male Idu (c.2150 BC) (Koller et al. 1998, p.343-344; Weser et al. 1998, p.511A-516A). Samples were found to contain a prevalence of fatty acids and steroids, in addition to hydrocarbons originating from the paraffin wax applied to the mummy when it was first discovered in the early 20th century. More notable were the cyclic alcohols, apparently derived from the wood tar supposedly employed in the mummification process, and the coniferous diterpenoids which were also detected. Normally minor components of fresh resin, methyl esters of the diterpenoid acids were relatively abundant, suggesting that the coniferous resin had been subjected to some form of heating process (Weser et al. 1998, p.512A). Pitch components such as retene were absent however. Oxidised diterpenoids were substantial, although their significance for the heating process is unclear. Whilst the results are certainly interesting given the relatively early date, the comment that "no conservation, according to general knowledge, was assumed to have been done" during the Old

Kingdom is simply not true, since there is ample evidence to the contrary (see Chapter 1 above).

A number of samples taken from a Third Intermediate Period mummy (c.700 BC) by M. Serpico and R. White were analysed using GC/MS analysis (Serpico & White 1998, p.1044). A sample taken from the thoracic cavity was found to contain dehydroabietic acid and 7-oxodehydroabietic acids, and retene was also present in significant abundance, pointing to a coniferous pitch. The absence of any degradation products of cis-abienol was used to exclude the possibility of it being a fir pitch, and it was concluded that the sample was a cedar or pine pitch (Serpico & White 1998, p.1044). The second sample was identified as pistacia pitch. Following further analysis, beeswax was also identified as a component of a "black, glossy, hardened deposit" taken from the thoracic cavity, in addition to the conifer pitch (Serpico & White 2000, p.467). Another sample taken from the back of the skull also consisted of a coniferous pitch, the same diterpenoid components being present (Serpico & White 2000, p.467). Beeswax was also present in trace amounts, and most notably small amounts of moronic and oleanonic acids were identified, confirming the presence of a pistacia resin (Serpico & White 2000, p.467). A sample taken from a canopic jar was also analysed, the detection of dehydroabietic acid and 7-oxodehydroabietic acid revealing the presence of a *Pinaceae* resin (Serpico & White 1998, p.1041; Serpico & White 2000, p.468).

The analysis of thirteen 'balms' taken from unprovenanced mummies dating from 1100 BC – AD 400 was carried out in 1999 by Connan using GC/MS (Connan 1999, p.33-50). All were found to contain a conifer resin and ten contained a fat, although this may have originated from the body itself. Beeswax and bitumen were also found in abundance, being present in six and eight of the 'balms', respectively, the sources for bitumen identified as both the Dead Sea and the area of modern Iraq. There were no obvious trends discernable in this study, all four commodities found being a major component of at least one of the samples analysed.

In 1999, a preliminary report of work described in this thesis of samples from two dynastic Egyptian mummies, Khnumnakht of the XIIth dynasty (c.1900 BC) and Horemkenesi of the XXIst dynasty (c.1050 BC) (Buckley et al. 1999, p.443-452). By employing GC/MS together with sequential thermal desorption-gas chromatography-mass spectrometry and pyrolysis-gas chromatography-mass spectrometry, the XIIth dynasty sample revealed fatty

acids and steroids indicative of an animal fat (although this may have derived from the body itself). This sample also revealed diketopiperazines deriving from a proteinaceous material. The sample from the XXIst dynasty mummy Horemkenesi consisted essentially of free fatty acids with a C_{16:0}/C_{18:0} ratio indicative of a plant derived oil or wax. This last finding contradicts the conclusions made from previous reported findings of tree resin (Taylor 1995, p.92).

In the most recent report to date, an Italian team analysed the 'balm' of a mummy from the Saite period (seventh century BC) (Columbini et al. 2000, p.19-29). A portion of 'resinous' mass from the left side of the thorax was analysed by GC/MS, its major component being pistacia resin identified by the presence of the triterpenoids moronic, oleanonic, isomasticadienonic and masticadienonic acids. The major triterpenoid however was norolean-17-en-3-one suggesting that the resin had been strongly heated. Lesser amounts of an unidentified vegetable oil (fatty acids) and beeswax (*n*-alkanes and wax esters) were also identified, in addition to a minor bitumen component (steranes and hopanes) (Columbini et al. 2000, Fig.7, p.26).

2.4.1 Summary of previous work

Having reviewed the results of previous chemical investigations it is appropriate to further summarise the current state of research into the organic materials employed in ancient Egyptian mummification, taking account of the analytical methods employed and samples selected for investigation.

Initial studies by Lucas, Reutter, Spielmann, Griffiths and Zaki and Iskander relied largely on tests of solubility, fluorescence and acid/saponification values, which along with a number of 'spot' tests involving characteristic colour changes were the main analytical techniques available at that time. Although their results are interesting, their necessary dependence upon essentially non-specific tests makes the findings obtained of limited value. The amorphous organic mixtures analysed cannot be satisfactorily investigated using such non-specific techniques susceptible to interferences (e.g. abietic acid test). Both Lucas and Griffiths acknowledged the limitations of the chemical techniques available to them, being particularly appreciative of the problems encountered when attempting to compare fresh reference materials with aged, often highly degraded (e.g. oxidised, polymerised) samples. Yet given that Lucas' reports are often the main source of

information for provenanced and dated mummies, the limitations of his techniques are particularly unfortunate.

In order to identify the complex, organic mixtures in question, it is desirable to employ the analytical techniques which will provide information at a molecular level to identify the individual components (the so-called biomarker compounds) present. In this way compounds characteristic of particular substances which have survived the degradation processes can be used to identify the original sources of the organic materials. GC/MS and LC/MS are particularly suited to this purpose and have been employed to this effect (Table 2.2). Yet of the studies which have utilised these techniques, only five have involved provenanced and dated mummies (Koller et al. 1998; Rullkötter & Nissenbaum 1988; Connan & Dessort 1991; Buckley et al. 1999; Columbini et al. 2000). Three of these studies involve the analysis of a single mummy, with the other two each including two provenanced and dated mummies (Connan & Dessort 1991; Buckley et al. 1999).

Although the subject of mummification holds undeniable fascination, it is only through the systematic study of dated, provenanced samples that a meaningful picture of the materials employed can begin to emerge. Yet given the undated and unprovenanced nature of the majority of the samples so far examined, the amount of useful information obtained remains meagre at best. The problem is further compounded by studies of Graeco-Roman mummies which attempt to understand pharaonic practices, something which is simply not possible given that the Ptolemies and their Roman successors inevitably transformed traditional Egyptian practices to reflect their own classical values.

Of the many hundreds of provenanced and dated mummies which have survived from the dynastic period (pre-332 BC), only four have been the subject of chemical investigations capable of identifying the aged nature of the complex organic materials used in their preservation, namely the Old Kingdom mummy 'Idu', (Koller et al. 1998), the Middle Kingdom 'Khnumnakht' and Third Intermediate Period 'Horemkenesi' (Buckley et al. 1999) and the Saite 'Merneith' (Columbini et al. 2000).

It is also notable that studies continue to use the terms 'resin' and 'bitumen' quite indiscriminately, with little consideration for the true nature of these organic materials (Aufderheide et al. 1999, p.197-210). Given their potential for illuminating otherwise darkened corners of ancient Egyptian culture involving subjects as diverse as trade routes,

technology and ritual practice, it is particularly unfortunate that this remains the case even amongst Egyptologists, including those specialising in the study of mummification itself.

2.5 AIMS AND OBJECTIVES OF THIS THESIS

As discussed above (Chapters 1 and 2), relatively little is known about the nature and origin of the organic embalming agents employed in mummification, despite repeated claims to the contrary. This is due to the virtual absence of systematic provenanced and dated studies carried out on Egyptian mummified remains to date. The investigation presented in this thesis uses a series of complementary analytical techniques (see Chapter 8) to determine the chemical composition of these complex embalming materials.

In this way the research aims to:

- i) elucidate the nature of these embalming agents, thus determining the origin of these substances.
- ii) to first develop and then assess the merits of a virtually non-destructive technique (i.e. sequential TD-GC/MS and Py-GC/MS, see Chapter 3) as an analytical approach attractive to museum curators, Egyptologists and archaeologists.

This thesis assesses the relative merits of the analytical techniques and then applies these to the characterisation and identification of:

- i) a significant number of samples from an individual mummy to assess the number and nature of the commodities employed in a single mummification.
- ii) samples from a significant number of human mummies (i.e. fourteen) in order to allow chronological comparison of the organic embalming agents employed in their preservation.
- iii) samples from a number of animal mummies in order to allow a comparison of the organic embalming agents employed in the preservation of the animals, and with the human mummies also investigated in this thesis.

CHAPTER 3

Analytical approach

CHAPTER 3: ANALYTICAL APPROACH

3.1 General background

Extensive chemical analysis is the only way to fully ascertain the nature of the materials (i.e. the organic embalming agents) used during the mummification process, their precise identification being only possible through the use of the most stringent scientific techniques. Yet far too much continues to be based on the assumptions and guesswork of those who examined mummies from a purely anatomical perspective, a problem compounded by the fact that many were studied on a single occasion, using highly destructive methods of examination, i.e. unwrapping. The rapid deterioration of the mummies since their discovery and/or unwrapping, combined with 'insensitive handling' (e.g. Petrie 1887-1889 in Drower 1985, p.96; Derry 1942 in correspondence, personal communication, J. Fletcher, etc.) and volatile political situations on both personal and international levels makes further studies of many Egyptian mummies highly unlikely.

Of those remaining mummified remains which can be studied, non-destructive techniques of examination obviously provide an invaluable way forward. Although they cannot identify the precise nature of the materials used in mummification, non-invasive techniques of medical imaging can nevertheless detect the presence of substances such as resin, which show up as a radiopaque material within the body and/or wrappings. The development of such techniques has obvious merit in the examination of Egyptian mummies, which no longer need to be unwrapped and potentially destroyed in order to study them. Indeed, the first mummies to be examined by X-ray occurred only three years after Röntgen's initial discovery, when Petrie undertook radiological examinations in 1898. Smith X-rayed the pharaoh Tuthmosis IV in 1903, with further studies conducted in Egypt by Derry throughout the 1920s-1940s, culminating in the systematic radiographical examination of all the royal mummies by an American-led team (Harris & Weeks 1973; Harris & Wente 1980). Radiographical techniques were also adopted to study Egyptian mummies in collections across the world, one of the most comprehensive studies initiated by the Boston Museum of Fine Arts in the 1930s and continuing into the 1980s, where further developments in non-invasive medical imaging led to the use of CT scans to provide ever more detailed results (Boston 1988, p.246).

Yet despite their tremendous value in many areas (e.g. the study of diseases, certain, morphological aspect of mummification), such non-destructive techniques of examination cannot answer questions concerning the nature and origin of the so-called 'resinous' materials these techniques can often reveal. The amorphous organic residues which represent the organic preservatives, perfumes and unguents can only be positively identified by their characterisation at the molecular level via chemical analysis, and it is only their molecular 'fingerprint' which has the potential to reveal the true nature and origin of these otherwise elusive 'balms'. It should also be noted that differentiation is by no means as straightforward as is generally perceived by both archaeologists and chemists. Certain sources can, especially after degradation, have a very similar chemistry to a completely unrelated source (e.g. the cinnamic acids present in aged storax and cassia/cinnamon; cadinenes in degraded cassia and myrrh). In both of these examples, there are indeed other biomarkers, i.e. triterpenoids, which could be expected to survive, potentially allowing a distinction to be made. Yet the degraded triterpenoids likely to be present can chemically resemble other commodities which may also have been utilised, such as a highly oxidised *Pistacia* resin. This is particularly so in the context of mummies, where highly oxidised species can often be prevalent. Misidentification is a real possibility, potentially leading to a complete misinterpretation of the archaeological evidence. Therefore a combination of a thorough approach and an open mind are crucial if these complex, degraded materials (quite possibly as mixtures) are to be successfully recognised.

3.2 Contamination

During the 19th Century it was not uncommon for false mummies to be produced by the local Egyptian inhabitants to meet the demand of European travellers who felt that their visit was not complete without a mummy to take home with them as a souvenir. Given that current museum collections are largely based upon donations from private collectors, this point is not unimportant, and illustrates the need for a provenance and date if studies are to be of maximum value. When it has been established that a mummified body is genuine, there remain a number of important considerations regarding the integrity of their chemical investigation. Contamination can potentially originate from a number of different sources, including that resulting from the transportation of mummies from Egypt to their eventual destination. Accidental contamination from substances such as nicotine and other drugs must be considered, given the vogue for 'mummy unwrapping parties' as an evening's entertainment amongst wealthy Victorians. The use of preservatives during this period has

also been noted, with varnishes, etc. having being applied in certain cases to 'preserve the preserved'! (e.g. Melville 1995, p.77-84). The potentially resinous nature of these substances could clearly lead to erroneous conclusions regarding the nature of the materials employed in embalming, particularly when records on any treatments carried out are unlikely to have been kept at that time. Conservation treatments carried out more recently must also be considered, although relevant records are more often available and can determine whether chemical analysis is likely to be problematic. The mummy of Horemkenesi, for example, had been treated with methyl bromide to fumigate the body, although the use of this gaseous substance would not be expected to cause significant problems for any subsequent analysis.

In addition to conservation treatments within museums, there are also potential problems associated with both previous and current excavation practices. It was not uncommon for mummies to be 'conserved' with paraffin wax at the excavation site, particularly the early part of the last century. The mummy of Tutankhamun was in fact treated in this manner (Carter 1927, p.149), as was the Old Kingdom/First Intermediate Period mummy of Idu, chemically analysed recently (Koller et al. 1998, p.343-344). If no records are kept, the analysis can clearly prove problematical, and even if its use is documented the resulting contamination can make investigation difficult, potentially obscuring any embalming agents which may have been employed.

The casual use of sample containers is also another area of concern, with early archaeologists having used recycled food packets and tea chests to hold artefacts including human remains. W.M.F. Petrie, considered by many to be the first archaeologist to have used a systematic and scientific approach to excavation, is a prime example (Drower 1985, p.96, 136 etc.). Clearly, residues from these containers have the potential to contaminate the remains of these mummies, thereby misleading scientists and Egyptologists as to the actual significance of the components detected in the chemical investigations. Even now it is far from unusual for freshly-excavated specimens to be collected and stored long-term in everything from cigarette packets to used food containers. Given the controversy surrounding the 'finding' of nicotine in Egyptian mummies (Balabanova et al. 1992, p.358) this, in addition to the habit of 19th Century pipe-smoking Victorian gentlemen undertaking mummy unwrappings for both 'scientific' and entertainment purposes, must cast some doubt on the apparent identification of strange and exotic substances found in mummies.

Clearly these factors can result in vital information being lost, making any subsequent analysis difficult and potentially misleading.

The sampling of mummies must necessarily be pragmatic, given their inherently sensitive nature. Where possible however, surface contamination and chemistry must be considered. If samples can be taken from areas of the body less likely to have been exposed to contamination, then obviously this is desirable. Obviously the outer surface areas of the body are more likely to have become contaminated, given their greater exposure. Surface areas are also more likely to have undergone significant chemical changes such as oxidation and hydration together with polymerisation, given the increased exposure to UV radiation which catalyses cross-linking of many organic materials. These highly polar, chemically-altered compounds are not only less amenable to analysis due to their increased polarity/insolubility/intractability, but are generally less easily characterised, since they are rarely encountered and therefore reported in the literature, and the preparation of these polyfunctionalised species in the laboratory may prove problematic. Relatively large sample sizes can of course obviate many of these problems, providing material which has largely been unexposed (with only a small portion of surface matter). The constituents of these samples are likely to be less polar/insoluble, given the relative lack of oxygen and UV radiation necessary for many of the degradation reactions to occur.

Yet, given the sensitive nature of the mummies concerned, large sample sizes are rarely possible nor practicable. Ideal circumstances would involve taking a substantial number of samples from as wide a range of anatomical locations as possible (including hair which is often overlooked), with duplicate samples taken to allow meaningful chemical comparisons to be made – an especially important factor where sample sizes are limited. Sample storage is also important, with the exclusion of light, moisture and oxygen the most important factors, although many mummies have been/are stored at temperatures close to room temperature, one museum collection being formerly located above a boiler house. Hence, there is often little to be gained by refrigeration or freezing; if low temperature storage is available this can be useful, but the major considerations should be the absence of light, moisture and oxygen.

3.3 ANALYTICAL TECHNIQUES

3.3.1 General considerations

When deciding upon an appropriate analytical approach, it is first necessary to consider both the valuable nature of the ancient specimens from which the samples are to be taken, and the specific nature of those samples (i.e. aged organic materials of uncertain origin). Due to their irreplaceable origin (i.e. mummified remains), the ability to accommodate very small sample sizes is an important consideration, particularly since a large number of mummies need to be studied to provide a comprehensive and meaningful picture of the embalming materials employed. Furthermore, the approach adopted must recognise the nature of the samples analysed, taking account of the fact that the compositions of the organic materials used in the mummification process are likely to have changed substantially over time through the natural processes of degradation. Such processes will include oxidation, reduction, hydrolysis, aromatisation and polymerisation. Yet the organic components which are resistant to chemical and biological degradation and are characteristic of the original 'embalming resins' can be expected to survive for very long periods of time and be recognisable in a relatively unchanged state. These specific compounds represent the biomarker components that will be used to identify the ancient 'resins'. The possibility of encountering both free and polymerised biomarker lipids must be considered when deciding upon the analytical approach to be taken.

3.3.2 GC/MS

Solvent extraction followed by GC-MS is often employed as an appropriate approach for the characterisation and identification of a wide variety of organic components, an approach which can provide a great deal of valuable information regarding the nature of aged, organic residues (Rullkotter & Nissenbaum 1988, p.618-621; Gulaçar et al. 1989, p.61-72; Koller et al. 1998, p.343-344; Evershed 1990, p.139-153). Following suitable derivatisation, even the polar polyfunctional compounds commonly present are amenable to GC-MS (Gulaçar et al. 1989; Evershed 1990, p.139-153). Nevertheless, sample preparation is relatively time consuming and sample losses, particularly of volatile components which may be trapped within the sample matrix (Evershed et al. 1997, p.432-433), can be problematical.

3.3.3 Sequential thermal desorption-gas chromatography/mass spectrometry (TD-GC/MS)

The technique of thermal desorption coupled with gas chromatography-mass spectrometry has proven to be a rapid and direct method for the identification of free biomarkers in a broad range of organic materials (e.g. Stott & Abbott 1995, p.227-237). Thermal desorption is time efficient, since the extraction step is effectively instantaneous. Furthermore, the technique involves minimal sample manipulation, reducing the problems of contamination, sample loss and other experimental errors associated with 'wet chemical' procedures. The very small sample size (<0.1 mg) allow a virtually non-destructive analysis of precious specimens (e.g. mummified remains). TD can also be conveniently combined sequentially with Py-GC-MS. The combination of TD- and Py-GC/MS is particularly attractive since this combined approach allows the study of both 'free' and 'bound' lipid biomarkers thought to be present in organic embalming agents.

TD-GC/MS also has the advantage of detecting any volatile components, which may have been trapped within the sample matrix, since no extraction or solvent evaporation procedures are involved. These relatively elusive components are a valuable source of chemical information, and since many of these are characteristic of a number of important commodities believed to have been used (e.g. volatile aromatics in spices; sesquiterpenoids in spices, myrrh, cedar and Pistacia resin), their potential presence should be considered. Furthermore, Py-GC/MS can detect the molecular building blocks of both polysaccharides and proteins, and since these are not observed using the more conventional solvent extraction approach, the sequential TD/Py serves to complement the solvent extraction procedures utilised. It should also be noted that although the small samples sizes can only provide a 'snapshot' of the materials present, the positive identification of frankincense for example (still not identified in an Egyptian mummy despite the common association of this commodity with the practice of embalming), would be of major importance regardless of how extensively it had been applied to the body.

3.3.4 Alternative analytical techniques

Other techniques which have been used to analyse the organic materials from Egyptian mummies include infrared [(IR; including FTIR) e.g. Storch & Schäfer 1985, p.328-328] and nuclear magnetic resonance [(NMR) e.g. Wallgren et al. 1986, p.321-327]. Both these

techniques can provide useful information at the molecular level, and indeed are powerful techniques for the characterisation of individual molecules (although relatively large sample sizes are required for both IR and NMR). However, they are of limited use when applied to the characterisation and identification of complex organic mixtures, and lack the specificity to chemically characterise and identify the degraded complex mixtures present in Egyptian mummies. It is only via the initial separation of individual components of these complex mixtures, followed by their subsequent characterisation and identification of the isolated biomarkers, that the unambiguous identification of these unknown degraded complex mixtures can be achieved. Although IR (or FTIR) was considered as an additional source of chemical information, with no extraction process necessary (thus limiting potential contamination), it was not in fact utilised due to both time constraints and the likelihood of 'natron' being present in these samples which would (without carrying out necessary extraction procedures) have resulted in the carbonate dominating the spectra, thereby limiting the value of the information.

3.4 Reference materials

Reference materials were not included in this research given the wide diversity of the commodities likely to have been employed. Some were indeed analysed to provide corroborating evidence for some of the findings, but given the time constraints of this study, they were not the subject of detailed chemical investigation. Those which were the subject of limited investigation included cedar, mastic, frankincense, myrrh, storax, henna, acacia, cinnamon, juniper and beeswax.

3.5 Summary

Only when all source materials have been fully examined can it be known for certain which materials were used by the ancient Egyptians to mummify their dead, from the tantalising clues given by the Egyptians themselves, the later accounts of classical authors and above all serious chemical studies, the most informative source of all.

CHAPTER 4

Horemkenesi: the systematic chemical study of an individual human mummy

CHAPTER 4: HOREMKENESI - THE SYSTEMATIC CHEMICAL STUDY OF AN INDIVIDUAL HUMAN MUMMY

4.1 OBJECTIVES

This chapter involves the in-depth chemical investigation of the mummified body of the XXIst dynasty (1069-945 BC) Theban priest Horemkenesi (Ill.7), in an attempt to chemically characterise and identify the organic materials employed in the embalming of an individual mummy. A significant number (15) of samples of various types (packing, wrappings, tissue, 'resin/bitumen'), taken from various anatomical locations, were analysed in order to obtain an overall picture of the materials used over the whole body. GC, GC/MS and sequential TD-GC/MS and Py-GC/MS were utilised to facilitate the characterisation and identification of the organic embalming agents employed. Initially, sequential TD-GC/MS and Py-GC/MS will be discussed, followed by results from the HT-GC/MS following chemical treatments and/or solvent extraction procedures (total lipid extract, acid fraction and neutral fraction) and derivatisation. Finally, a discussion of degradation pathways, in particular oxidative changes, will conclude this chapter.

4.2 INTRODUCTION

Of the few studies carried out to determine the organic embalming agents employed in ancient Egyptian mummification (see 2.4), using the methods of chemical analysis (GC/MS, etc.) capable of identifying such complex degraded residues, none have analysed more than two samples from an individual provenanced and dated mummy. Although these findings are interesting, the standard approach of analysing only one or two of these amorphous organic materials can in no way be said to give an accurate and meaningful picture of the embalming agents employed. It is only by the thorough, systematic study of an individual mummy, with a significant number of samples taken from a range of contexts, i.e. packing, wrapping, tissue, 'resinous' material, etc., with anatomical considerations taken into account that an accurate and meaningful picture can begin to emerge.

This is crucial if an overall picture of the materials used on different parts of the body is to be obtained, given the variation in materials described in the ancient texts (e.g the Ritual of

Embalming; see Chapter 1). Perfumed material used in the packing of the thorax, 'resin'/'wax' to cover the embalming incision, and the particular attention paid to eyes, ears, lips and nose may all indicate that these areas were treated in an alternative manner to other parts of the body. Yet without a rigorous approach to sampling, the understanding of the materials employed will necessarily be limited which in turn will limit the understanding not only of ancient Egyptian mummification but areas as diverse as trade links, symbolism, etc. Indeed, the importance of a multiple sampling approach was demonstrated by Serpico and White (Serpico & White 1998, p.1037-1048).

The study presented here examined 15 samples from the provenanced and dated dynastic mummy Horemkenesi (see Table 4.1), his body prepared at a time (c.1050 B.C.) when the mummification process was considered to be of a generally high standard. Samples of packing, wrappings, tissue, 'resin' and whitish 'efflorescence' material were analysed. Care was taken to avoid potentially contaminated areas already exposed, and sample sizes taken reflected the need to obviate any problems of this nature, in addition to difficulties associated with highly oxidised/degraded material on the surface of the resins, waxes, fats, etc.

Diagnostic marker compounds present in the original embalming agents and resistant to degradation can be related to specific embalming agents and were therefore used to identify the 'balms'. GC/MS and TD/Py-GC/MS were used to facilitate the molecular separation and identification of these marker compounds. Due to the nature and history of the proposed embalming materials both free and polymerised components were likely to be present (see Chapter 2). Therefore the 'dual' approach of GC/MS (following solvent-extraction procedures) and sequential TD-GC/MS and Py-GC/MS was employed to allow the characterisation and identification of both the free (solvent-extractable) marker compounds and the recognisable sub-units of polymeric materials not amenable to the more conventional GC/MS approach.

4.3 BACKGROUND

The potential origin of the organic embalming agents has been discussed above (Chapter 1), together with the archaeological and historical background of these materials. In summary, true resins (conifer and *Pistacia*), gum (e.g. frankincense and myrrh) and balsamic (e.g. storax) resins, beeswax, bitumen, animal fats, plant oils and others (Tables

Table 4.1. Horemkenesi¹ (male adult), XXIst dynasty (c. 1069-945 BC), Third Intermediate Period, Thebes (Ha 7386): origin and nature of samples analysed.

Sample location and description ²	Sample type
Packing material (natron?) between wrappings and skin of left(?) upper forearm [9] ³ Ha7386/731	Packing
Packing material from leg and foot (left) [10] ³ Ha7386/856	Packing
Mud packing from thoracic cavity [11] ³ 7386/804	Packing
Packing material/wrapping from right calf [4] ³ 7386/760	Packing/wrapping
Piece of 'resin'-soaked wrapping from left ankle/talus [1] ³ H7386/839	Wrapping
Threads from wrappings from left ankle/talus [3] ³ H7386/839	Wrapping
Wrapping fragments [2] ³ Ha7386/890	Wrapping
Wrapping/tissue from right calf [5] ³ 7386/760	Wrapping/tissue
Muscle fibres from head of right femur [7] ³ Ha7386/945	Tissue
'Resinous' material/muscle tissue from left hip/base of spine [8] ³ Ha7386/948	'Resin'/tissue
'Resinous' material from left side of upper spine [15] ³ Ha7386/908	'Resin'
'Resin' around the mouth[12] ³ Ha7386/686	'Resin'
Whitish 'efflorescence' from head of right femur (a) [6] ³ Ha7386/945	?
Whitish 'efflorescence' from head of right femur (b) [17] ³ Ha7386/945	?
Whitish 'efflorescence' from head of right femur (c) [18] ³ Ha7386/945	?

¹ Bristol Museum; ² the term 'resin' denotes physical appearance and does not presuppose any chemical composition or biological origin; ³ museum number.

1.3, 2.1) are all possible sources of embalming materials. Yet the indiscriminate use of the terms 'resin' and 'bitumen' perpetuate the extreme ignorance surrounding the nature of the organic materials used in mummification, a situation the current study seeks to begin to rectify.

The diverse chemistry of the organic embalming materials has also been described above (Chapter 2, 2.3) and so will not be detailed here. The chemistry of each of the samples analysed will be dealt with in turn in the results section and related back to their likely origin. In general, degradation processes such as dehydration, aromatisation, polymerisation and particularly oxidation are likely to have taken place over the 3,000 years since the burial of Horemkenesi. Interpretations must be made with circumspection, given that mixtures may well have been used (Herodotus II, 83-90, 1954, p.160-161; Diodorus in Smith & Dawson 1924, p.62-63), and an open mind is a crucial prerequisite if the picture obtained is to be at all meaningful.

4.4 RESULTS

The focus of this study was the characterisation and identification of the organic embalming agents employed in each of the mummies investigated. A further important aspect of the research was to assess the value of sequential TD/GC-MS and Py-GC/MS which is of particular value given the small sample sizes required (~0.1 mg) and the limited sample preparation involved. Due to the desire to compare and contrast the chemical nature of the samples taken, each technique is reported in turn with the results of each sample being described and compared with other samples analysed. The findings obtained for a given sample will then be discussed, with the relative merits of the techniques utilised. A comparison between TD-GC/MS and the more conventional GC/MS will also be made with a discussion of the findings and the relative merits of the techniques employed, together with the implications of the information they have provided.

The majority of the 'total lipid extracts', where there was sufficient sample size available, were separated into 'acid' and 'neutral' fractions using aminopropyl Bond elut columns, before being derivatised with BSTFA. This fractionation was carried out due to the likely complex nature of the total lipid extracts, potentially resulting in the co-elution of many of the components present making the elucidation of these complex materials extremely difficult.

For a summary of the findings of this study see Table 4.2. The identification of the compounds observed was based on both their mass spectra (NIST/EPA/NIH Mass Spectral Database) and retention times. For TD/Py-GC/MS the compounds are present as the free compounds. The compounds identified in the solvent extracts (acid and neutral fractions) are present as the free compounds or as their TMS derivatives.

4.4.1 Thermal desorption-gas chromatography/mass spectrometry

TD-GC/MS was carried out using a thermal desorption temperature of 310°C for 10s.

4.4.1.1 Packing material (natron?) between wrappings and skin of left(?) upper forearm [9] (Ha7386/731)

The results of the TD-GC/MS analysis revealed a series of short chain monocarboxylic acids in the C_{5:0} to C_{9:0} carbon number range, with C_{6:0} predominating. No longer chain fatty acids (e.g. C_{16:0}) were present, even as trace components. Also significant constituents of the TD profile were the C_{8:0}, C_{9:0} and C_{10:0} 2-alkanones (characterised by fragment ions of m/z 58 and 71), the latter being the major compound detected, and a series of γ - and δ -lactones (characterised by base peaks m/z 85 and 99 respectively), i.e. C_{7:0} to C_{16:0} γ -lactones, with the exception of C_{13:0}, and C_{7:0} to C_{10:0} and C_{16:0} δ -lactones). These lactones maximised at C_{9:0}. No other components (other than contaminants, i.e. phthalate plasticizers) were observed.

4.4.1.2 Packing material from leg and foot (left) [10] (Ha7386/856)

The results of the TD-GC/MS analysis are shown in Figure 4.1a. The TD profile is dominated by a series of short chain monocarboxylic acids in the C_{5:0} to C_{9:0} carbon number range, with C_{7:0} predominating. The longer chain fatty acids, C_{16:0}, C_{18:1} and C_{18:0}, were not detected. Also significant constituents were a series of γ - and δ -lactones (C_{7:0} to C_{16:0}), maximising at C_{9:0} (of similar abundance to the C_{9:0} fatty acid) with no odd-over-even predominance. Of similar abundance, were the C_{8:0}, C_{9:0} and C_{10:0} 2-alkanones and the C_{7:0}, C_{8:0} and C_{9:0} aldehydes (m/z 43, 57, 70, 82 and 98). Minor amounts of the C₁₆ to C₁₉ *n*-alkanes (m/z 57 and 71), with no odd-over-even predominance, along with more appreciable quantities of the C₂₅, C₂₇ and C₂₉ homologues and the C_{16:0} nitrile (m/z 41, 43, 97, 110 and 124) were also observed.

Table 4.2. Horemkenesi¹ (male adult), XXIst dynasty (c. 1069-945 BC), Third Intermediate Period, Thebes (Ha 7386): origin and nature of 'balms' and their composition.

Sample location and description ²	Sample type	Inferred components of embalming "resin"	Relative abundance (%)
Packing material (natron?) between wrappings and skin of left(?) upper forearm [9] ³ Ha7386/731	Packing [P1]	Fat/oil (95% polymeric) ^{a,e,i,m,q P1}	100
Packing material from leg and foot (left) [10] ³ Ha7386/856	Packing [P2]	Fat/oil (90% polymeric) ^{b,i,j,n,f P2}	100
Mud packing from thoracic cavity [11] ³ 7386/804	Packing [P3]	Fat/oil (100% polymeric) ^{c,g,k,o,s P3}	100
Packing material/wrapping from right calf [4] ³ 7386/760	Packing/wrapping [P4]	Fat/oil (90% polymeric) ^{d,h,l,p,t P4} Wax	98 2
Piece of 'resin'-soaked wrapping from left ankle/talus [1] ³ H7386/839	Wrapping [W1]	Fat/oil ^{W1}	100
Threads from wrappings from left ankle/talus [3] ³ H7386/839	Wrapping [W2]	Fat/oil ^{W2} Wax	98 2
Wrapping fragments [2] ³ Ha7386/890	Wrapping [W3]	Fat/oil ^{W3} Wax	100 trace
Wrapping/tissue from right calf [5] ³ 7386/760	Wrapping/tissue [W4]	Fat/oil ^{W4} Wax	99.5 0.5
Muscle fibres from head of right femur [7] ³ Ha7386/945	Tissue [T1]	Fat/oil ^{T1} Balsam/umbelliferae?	99.5 0.5
'Resinous' material/muscle tissue from left hip/base of spine [8] ³ Ha7386/948	'Resin'/tissue [R1]	Fat/oil ^{R1} Proteinaceous material	100 trace
'Resinous' material from left side of upper spine [15] ³ Ha7386/908	'Resin' [R2]	Fat/oil ^{R2} Proteinaceous material	98.5 1.5
'Resin' around the mouth [12] ³ Ha7386/686	'Resin' [R3]	Fat/oil ^{R3} Proteinaceous material	100 trace
Whitish 'efflorescence' from head of right femur (a) [6] ³ Ha7386/945	? [Un1]	Fat/oil ^{Un1}	100
Whitish 'efflorescence' from head of right femur (b) [17] ³ Ha7386/945	? [Un2]	Fat/oil ^{Un2} Wax Proteinaceous material	97.5 0.5 2
Whitish 'efflorescence' from head of right femur (c) [18] ³ Ha7386/945	? [Un3]	Fat/oil ^{Un3} Wax Proteinaceous material	98 1 1

¹ Bristol Museum; ² the term 'resin' denotes physical appearance and does not presuppose any chemical composition or biological origin; ³ museum number; ⁴ % relative abundance based on absolute concentrations, calculated based on internal standards added at the extraction stage (TD taken into account where appropriate). Superscript letters (a-t) refer to histograms shown in Fig. 4.14. Superscript letters (P1-P4, W1-W4, T1, R1-R3 & Un1-Un3) refer to histograms shown in Fig. 4.15.

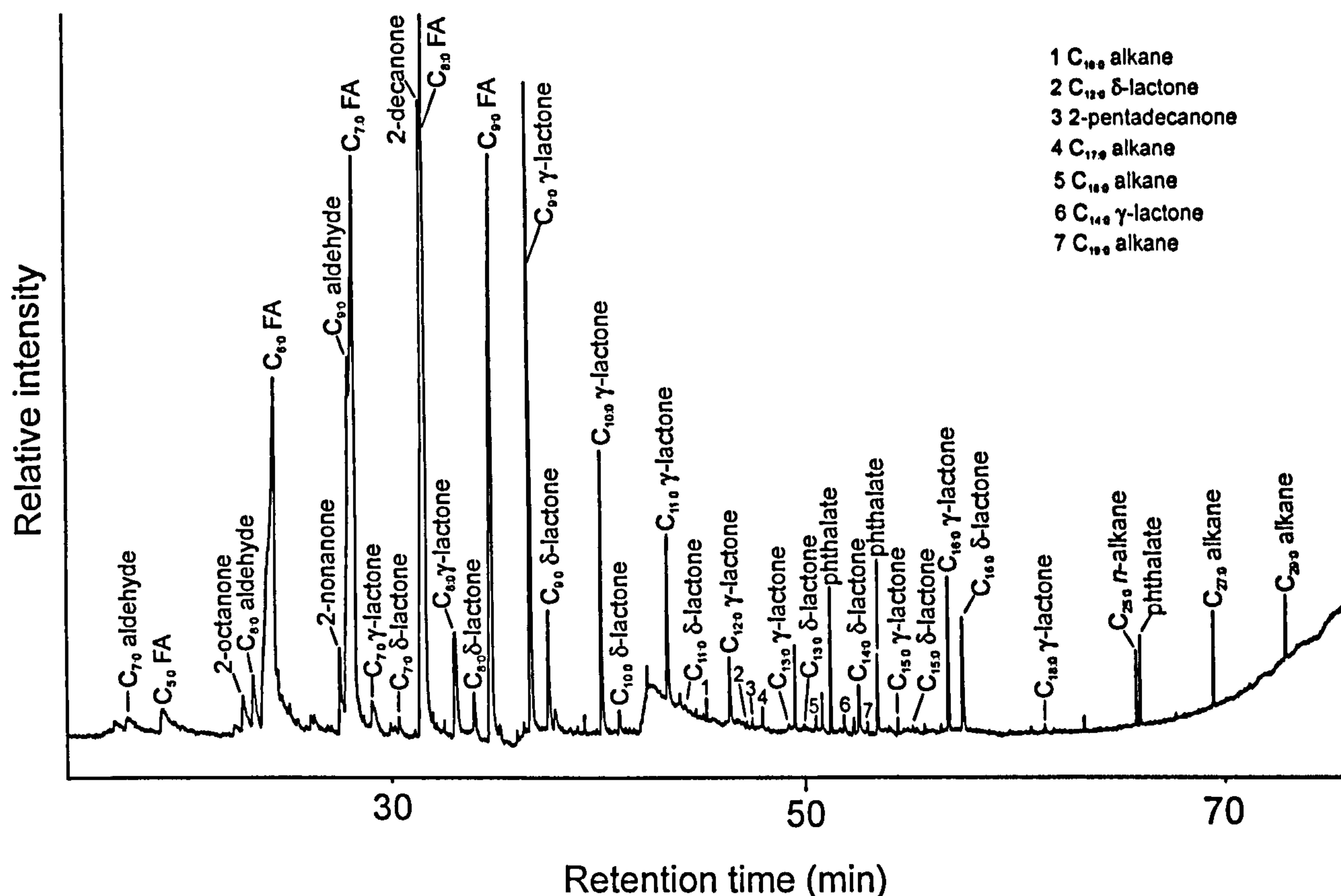


Figure 4.1a Total ion chromatogram of the thermal desorption profile (310°C/10s) of packing material from the leg and foot (left) [10] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

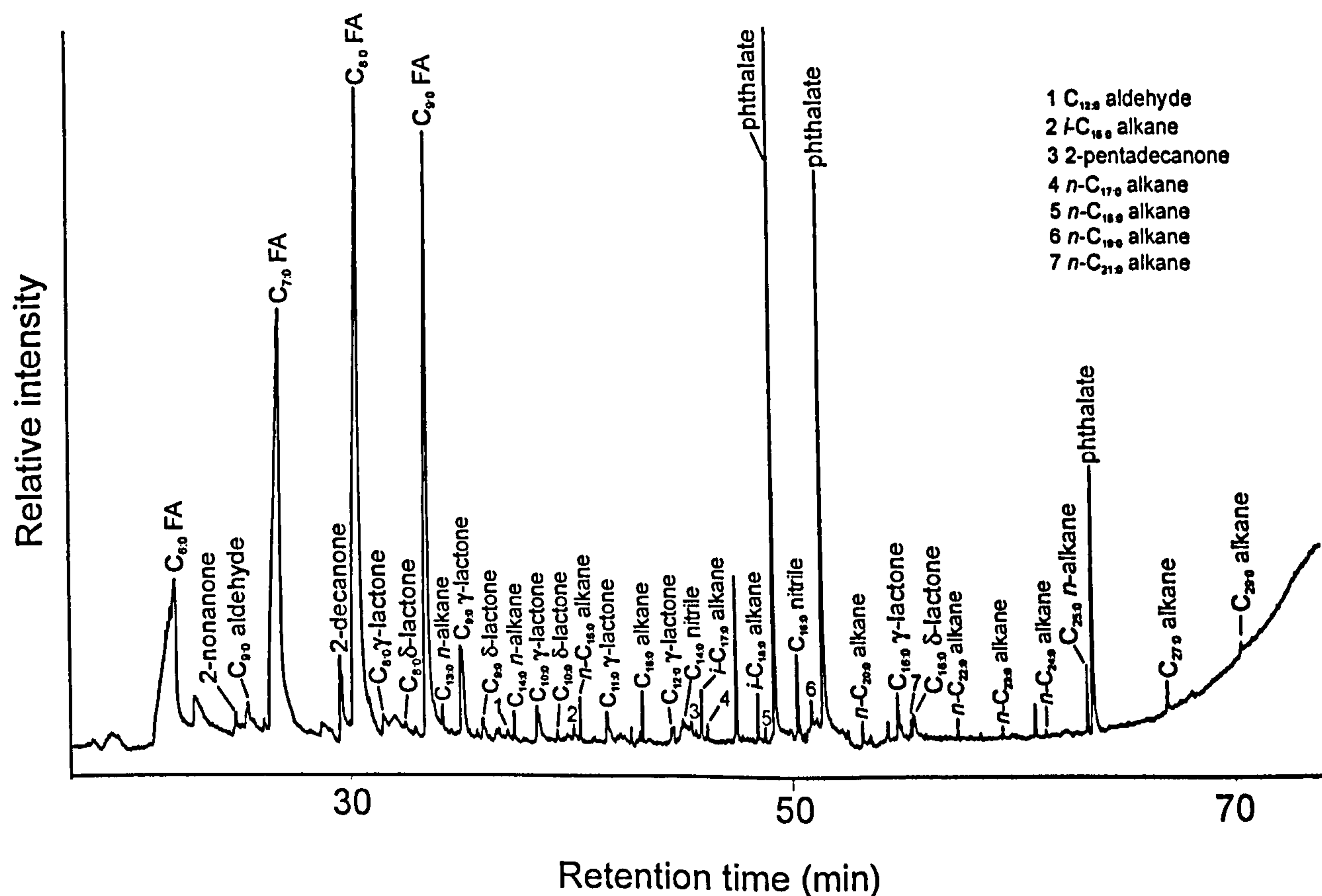


Figure 4.1b Total ion chromatogram of the thermal desorption profile (310°C/10s) of packing material/wrapping from the right calf [4] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

4.4.1.3 Mud packing from thoracic cavity [11] (7386/804)

The results of the TD-GC/MS analysis revealed no detectable components, indicating the absence of any free, thermally extractable lipids in this sample. It should be noted however, that highly polar material could have been present, which would not have successfully eluted from the column, or indeed volatilised sufficiently in the probe.

4.4.1.4 Packing material/wrapping from right calf [4] (7386/760)

The results of the TD-GC/MS analysis are shown in Figure 4.1b. The TD profile is dominated by a series of short-chain monocarboxylic acids in the C_{6:0} to C_{9:0} carbon number range, with C_{7:0} and C_{8:0} predominating. The longer chain fatty acids, C_{16:0}, C_{18:1} and C_{18:0} were not detected. Also significant constituents of the TD profile are a series of γ - and δ -lactones (C_{7:0} to C_{16:0}), maximising at C_{9:0} with no odd-over-even predominance. Minor amounts of the C₁₃ to C₂₄ *n*-alkanes, with no odd-over-even predominance, along with similar abundances of the C₂₅, C₂₇ and C₂₉ homologues, were also observed.

4.4.1.5 Piece of 'resin'-soaked wrapping from left ankle/talus [1] (H7386/839)

The results of the TD-GC/MS analysis are shown in Figure 4.2a. TD revealed palmitic (C_{16:0}) acid as the only major component. None of the short chain monocarboxylic acids (C_{6:0} to C_{9:0}), observed in the previous four samples, were detected. Minor amounts of myristic (C_{14:0}) acid, cyclohexanol and hexadecanal were the only other components identified. Interestingly, no furan or cyclopentenone derivatives were observed, indicating that intact cellulose does not produce these cellulose/sugar markers at the TD temperature (310°C) utilised in this study.

4.4.1.6 Threads from wrappings from left ankle/talus [3] (H7386/839)

The results of the TD-GC/MS analysis revealed a series of monocarboxylic acids (C₁₄ to C₁₈), the major components being C_{16:0}, C_{17:0}, C_{14:0} and C_{18:0} in decreasing order of abundance. Cyclohexanol was also present as a major component with appreciable amounts of short chain monocarboxylic acids in the C_{7:0} to C_{10:0} carbon number range, with C_{9:0} predominating. The C_{9:0} γ -lactone was also detected as a minor component, as were benzoic and hydrocinnamic acids and 6-methyl heptane-2,4-dione. Minor quantities of the C₂₅ to C₃₁ *n*-alkanes, with an odd-over-even predominance, were also observed. The observation of furan and 2-cyclopentenone derivatives, which were observed in moderate abundance, could suggest a possible sugar/plant gum origin, although given the nature of

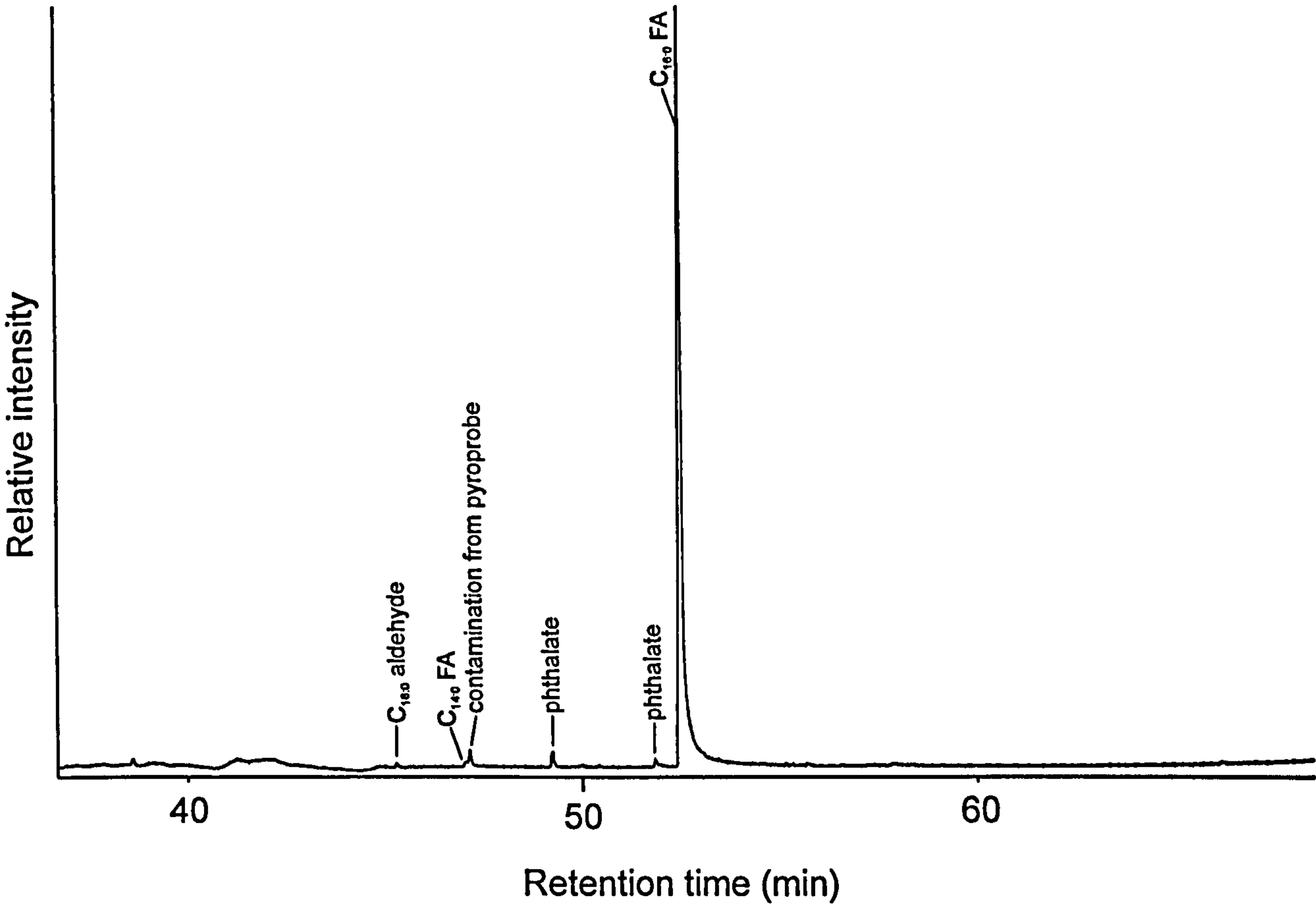


Figure 4.2a Total ion chromatogram of the thermal desorption profile (310°C/10s) of a piece of 'resin'-soaked wrapping from the left ankle/talus [1] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

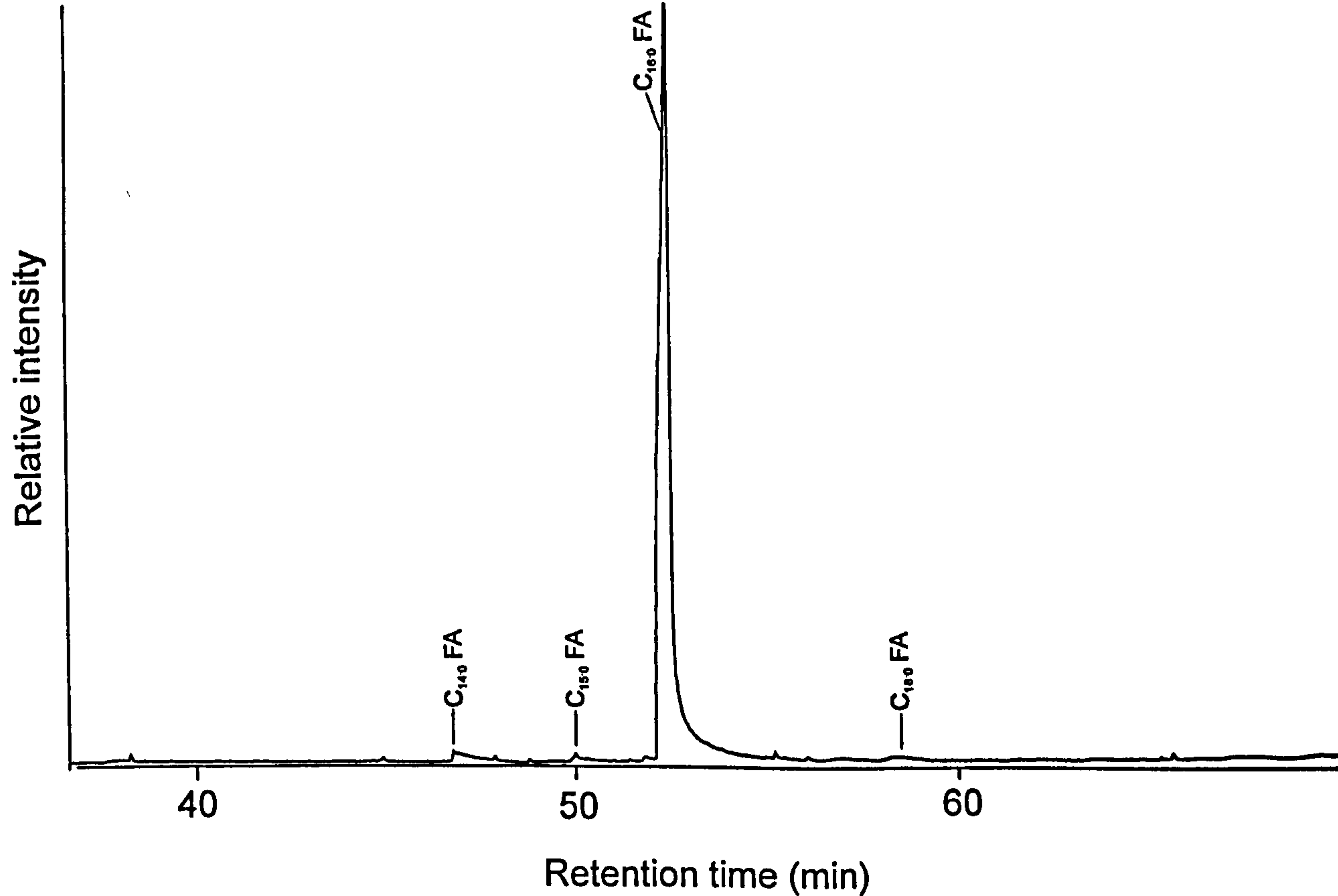


Figure 4.2b Total ion chromatogram of the thermal desorption profile (310°C/10s) of wrapping/tissue from the right calf [5] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

the sample these products may in fact derive from hydrolysed cellulose present in the degraded linen wrappings.

4.4.1.7 Wrapping fragments [2] (Ha7386/890)

The results of the TD-GC/MS analysis revealed palmitic (C_{16:0}) acid as the dominant component of the TD profile, with relatively minor amounts of the C_{14:0}, C_{16:1}, C_{18:1} and C_{18:0} fatty acids. In even lesser abundance were the short chain monocarboxylic acids (C_{7:0} to C_{9:0}) and the C₁₃ to C₁₇ *n*-alkanes, with no odd-over-even predominance, along with similar abundances of the C₂₅, C₂₇ and C₂₉ homologues. Other compounds noted were hexadecanenitrile ([M-15]⁺ 222) characterised by ions of *m/z* 110 and 124, and *n*-nonanal. No furan or cyclopentenone derivatives were observed, indicating that the sample does not contain degraded cellulose.

4.4.1.8 Wrapping/tissue from right calf [5] (7386/760)

The results of the TD-GC/MS analysis are shown in Figure 4.2b. TD revealed palmitic (C_{16:0}) acid as the only major component of the TD profile. No short chain monocarboxylic acids (C_{6:0} to C_{9:0}), observed in previous samples, were detected. Minor amounts of myristic (C_{14:0}), pentadecylic (C_{15:0}) and stearic (C_{18:0}) acids were the only other components identified. No furan or cyclopentenone derivatives were observed, indicating that the sample does not contain degraded cellulose.

4.4.1.9 Muscle fibres from head of right femur [7] (Ha7386/945)

The results of the TD-GC/MS analysis revealed palmitic acid as the dominant component of the TD profile, with C_{18:1} in moderate abundance and minor amounts of the C_{14:0}, C_{16:1}, and C_{18:0} fatty acids. No short chain monocarboxylic acids (C_{6:0} to C_{10:0}) were detected nor were any other significant components.

4.4.1.10 'Resinous' material/muscle tissue from left hip/base of spine [8] (Ha7386/948)

The results of the TD-GC/MS analysis displayed a series of monocarboxylic acids (C₁₄ to C₁₈), the major components being C_{16:0}, C_{18:1}, C_{18:0} and C_{14:0} in decreasing order of abundance. Minor constituents included *n*-nonanal and 2,5-diketopiperazine derivative pro-gly (*m/z* 70, 83, 111, M⁺ 154), in addition to the C_{15:0}, C_{16:1} and C_{17:0} fatty acids. Trace amounts of short chain monocarboxylic acids (C_{8:0} and C_{9:0}) were also detected.

4.4.1.11 'Resinous' material from left side of upper spine [15] (Ha7386/908)

The results of the TD-GC/MS analysis are shown in Figure 4.3a. The C_{16:0} fatty acid dominated the TD profile, with lesser amounts of the C_{18:0} and C_{14:0} monocarboxylic acids. Present as trace components were a number of 2,5-diketopiperazine and pyrrole derivatives and the C_{8:0} to C_{10:0} short chain fatty acids. The C_{16:0} and C_{18:0} amides characterised by *m/z* 59 and 72 were also identified as trace constituents.

4.4.1.12 'Resin' around the mouth [12] (Ha7386/686)

The results of the TD-GC/MS analysis are shown in Figure 4.3b. The C_{16:0} fatty acid dominated the TD profile, with moderate amounts of the C_{18:0} C_{18:1} and C_{14:0} monocarboxylic acids. Minor quantities of the C_{15:0} and C_{17:0} fatty acids were also observed. Two 2,5-diketopiperazine derivatives [including proline-glycine (pro-gly) (*m/z* 70, 83, 111, M⁺ 154)] were present as trace components, as were two C_{18:2} fatty acids, cholesta-3,5,7-triene and the C₂₅, C₂₇ and C₂₉ *n*-alkanes.

4.4.1.13 Whitish 'efflorescence' from head of right femur (a) [6] (Ha7386/945)

The results of the TD-GC/MS analysis revealed a series of monocarboxylic acids (C₁₄ to C₁₈) were detected, the major components being C_{16:0}, C_{18:1}, C_{18:0}, C_{14:0}, and C_{16:1} in decreasing order of abundance. Two C_{18:2} fatty acids were also observed as minor constituents.

4.4.1.14 Whitish 'efflorescence' from head of right femur (b) [17] (Ha7386/945)

The results of the TD-GC/MS analysis are shown in Figure 4.4. A series of monocarboxylic acids (C₁₄ to C₁₈) were detected, the major components being C_{18:1}, C_{16:0}, C_{16:1} C_{18:0} and C_{14:0} in decreasing order of abundance. The 2,5-diketopiperazine derived from pro-gly was present as a minor component, as was the C_{18:1} amide, two C_{16:2} fatty acids and two C_{18:2} fatty acids. Eluting at later retention times were the steroidal compounds cholesta-3,5,7-triene, cholesta-3,5-diene, cholesta-3,5-dien-7-one, and the C₂₅, C₂₇ and C₂₉ *n*-alkanes.

4.4.1.15 Whitish 'efflorescence' from head of right femur (c) [18] (Ha7386/945)

The results of the TD-GC/MS analysis displayed a series of monocarboxylic acids (C₁₄ to C₁₈), the major components being C_{16:0}, C_{18:1}, C_{14:0}, C_{18:0} and C_{16:1} in decreasing order of abundance. The aldehydes nonanal, undecanal and dodecanal were observed as significant constituents, with lesser amounts of the C_{8:0} to C_{10:0} short chain fatty acids. One C_{16:2} and

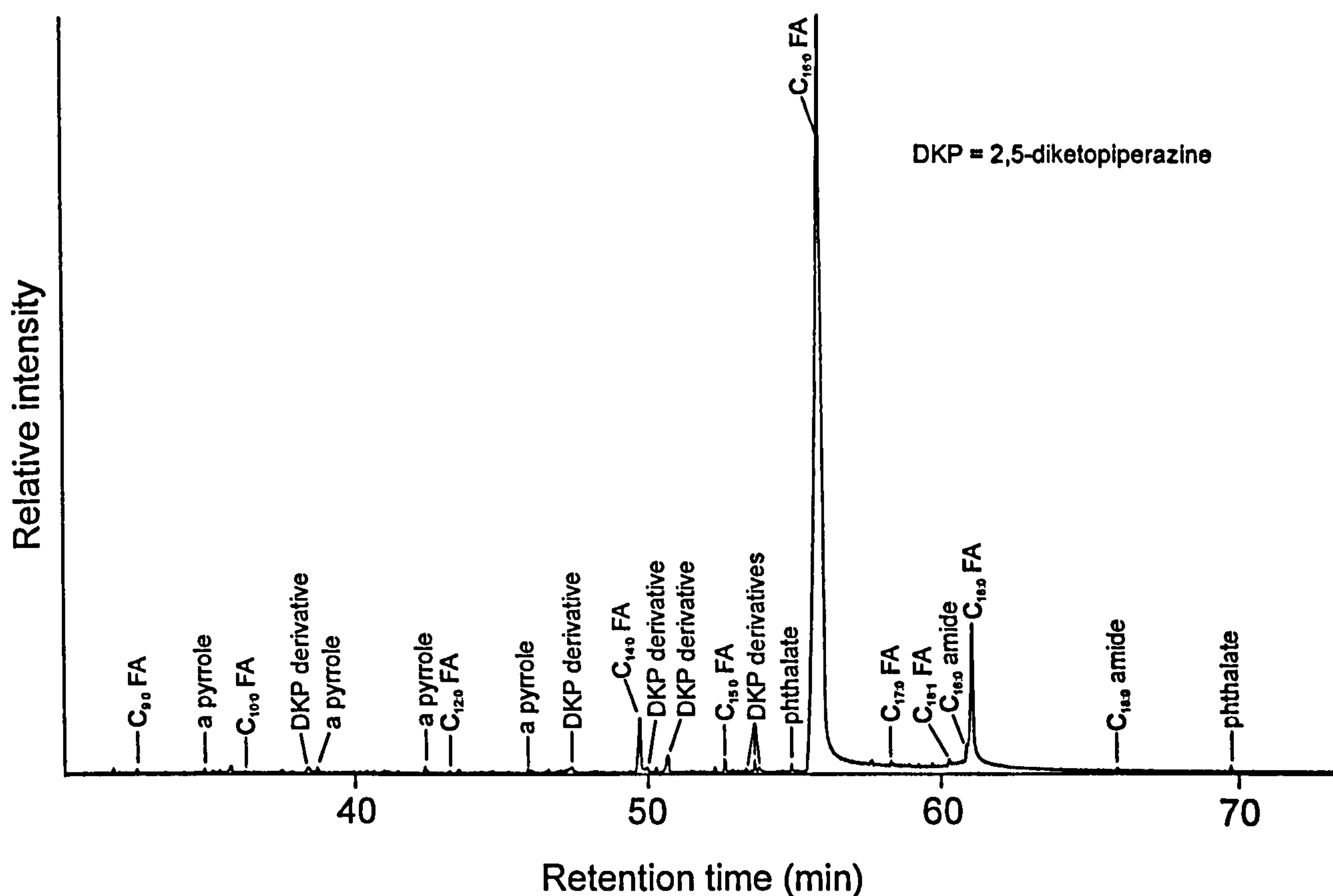


Figure 4.3a Total ion chromatogram of the thermal desorption profile (310°C/10s) of ‘resinous’ material from the left side of the upper spine [15] of the Theban priest ‘Horemkenesi’, XXIst dynasty (c. 1069-945 B.C.).

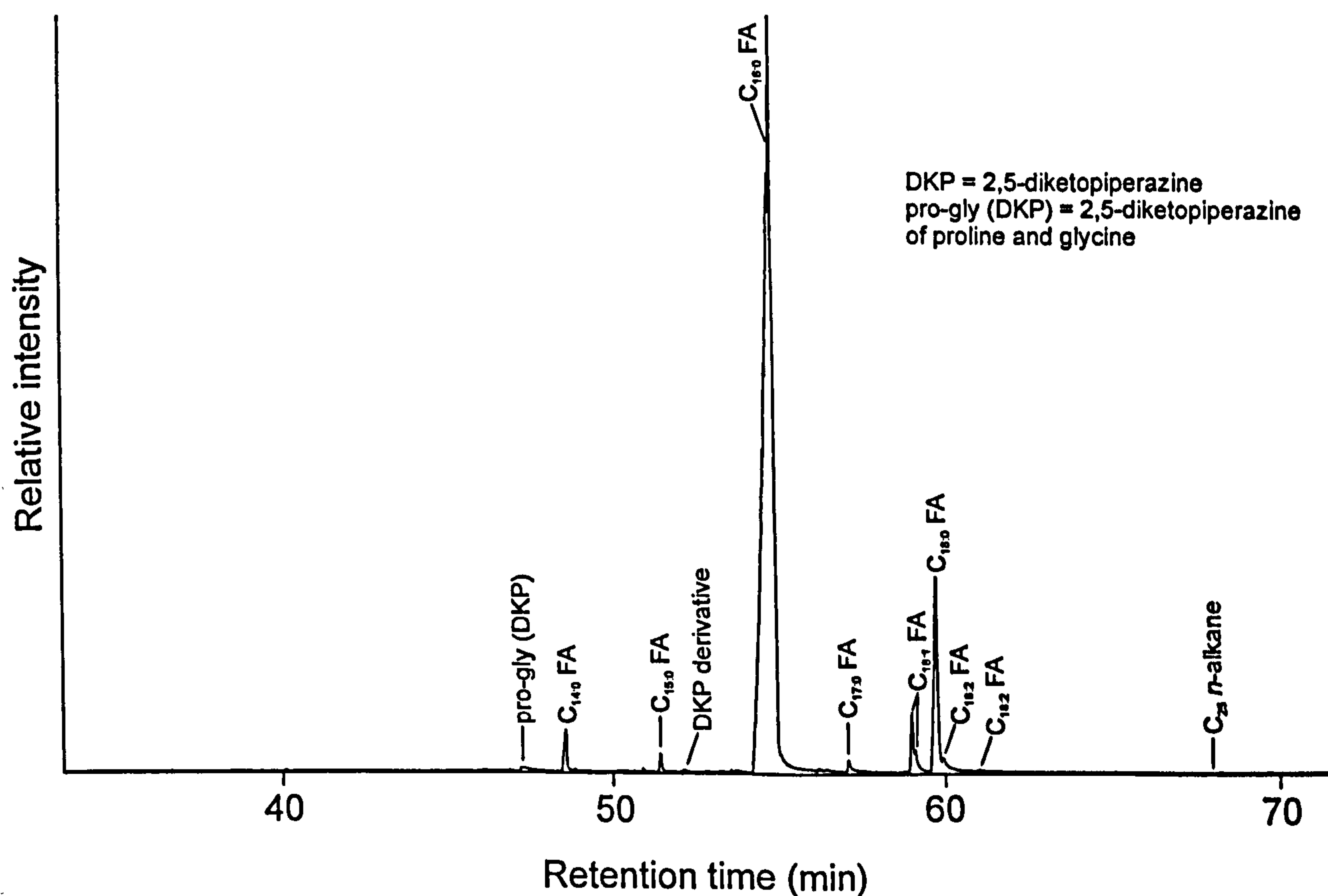


Figure 4.3b Total ion chromatogram of the thermal desorption profile (310°C/10s) of ‘resin’ around the mouth [12] of the Theban priest ‘Horemkenesi’, XXIst dynasty (c. 1069-945 B.C.).

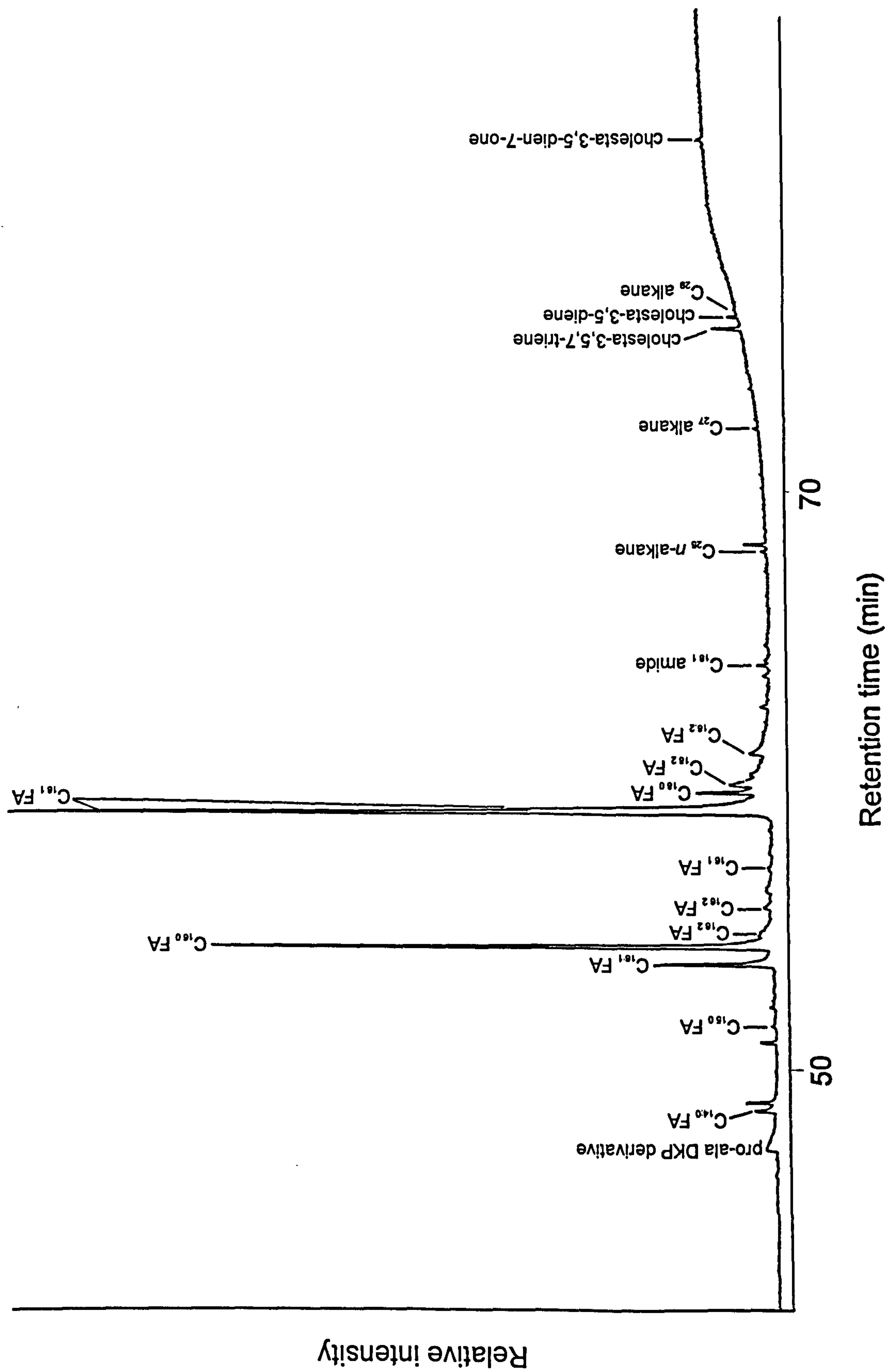


Figure 4.4 Total ion chromatogram of the thermal desorption profile (310°C/10S) of 'whitish effluorescence' from the head of the right femur (b) [17] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

two C_{18:2} fatty acids were present as minor constituents, as was a 2,5-diketopiperazine derivative and the C_{18:1} amide. Eluting at later retention times were cholesta-3,5,7-triene and cholesta-3,5-dien-7-one, and the C₂₅ to C₃₁ *n*-alkanes with an odd-over-even predominance.

4.4.2 Pyrolysis-gas chromatography/mass spectrometry

Py-GC/MS was carried out using a pyrolysis temperature of 610°C for 10s (after thermal desorption at 310°C for 10s).

4.4.2.1 Packing material (natron?) between wrappings and skin of left(?) upper forearm [9] (Ha7386/731)

The results of the Py-GC/MS analysis displayed a series of alkene/alkane doublets (m/z 55/57; C₆ to C₂₁), with the *n*-C₁₅ homologue most abundant. In lower abundance are the *cis*- and *trans*-2-alkenes in the C₈ to C₁₈ carbon number range and a number of alkadienes, including the 1,3-alkadienes (m/z 54 and 67; C₉ to C₁₅), a C₉ 1,4-alkadiene (m/z 67 and 81) and the 2,4-alkadienes (m/z 68 and 81; C₉ and C₁₂ and m/z 68 and 82; methyl branched *i*-C₁₀, *i*-C₁₁ and *i*-C₁₅). The major components were methyl ketones (C₆ to C₁₉), maximising at *n*-C₉ characterised by mass chromatograms m/z 58 and 71, along with lesser amounts of the more unusual ethyl (3-) ketones (m/z 57 and 72; C₁₀, C₁₁ and C₁₈). The saturated (m/z 71 and 86; C_{11:0} and C_{19:0}) and unsaturated (2-ene) (m/z 69 and 84; C_{19:1}) propyl (4-) ketones were present along with abundant mid-chain ketones (5- to 9-ones) in the C₁₀ to C₁₈ carbon number range, maximising at C_{15:0} with the co-eluting 7-pentadecanone (m/z 57, 71, 85, 113, 128, 141 and M⁺ 226) and 8-pentadecanone (m/z 57, 71, 99, 127, 142, 155 and M⁺ 226). At later retention times (58 to 72 min), long chain diketones were observed in moderate amounts, being identified as 4,12-octadecanedione (m/z 43, 58, 71, 85, 113, 129, 141 and [M-71]⁺ 211), 2,14-octadecanedione (m/z 58, 71, 85, 101, 113 and [M-43]⁺ 239) 2,13-octadecanedione (m/z 58, 71, 99, 115, 127 and [M-43]⁺ 239), 2,12-octadecanedione (m/z 58, 71, 85, 113, 129, 141 and [M-43]⁺ 239), 2,11-octadecanedione (m/z 57, 71, 99, 127, 143, 155 and [M-43]⁺ 239), 2,10-octadecanedione (m/z 57, 71, 113, 141, 157, 169 and [M-43]⁺ 239) and 2,9-octadecanedione (m/z 57, 71, 127, 155, 171 and [M-43]⁺ 239). Significant constituents of the pyrolysate included cyclic ketones (C₅ to C₇), alicyclic and aromatic hydrocarbons, aldehydes in the C₇ to C₉ carbon number range (characterised by m/z 43, 57, 70, 82 and 98), and the C_{8:0}, C_{16:0} and C_{18:0} nitriles (characterised by m/z 41, 43, 97, 110 and 124). The pyrolysate constitutes the majority

(>95% w/w) of the sample (i.e. *cf.* TD), indicating the presence of abundant bound lipids and pointing to a high degree of polymerisation.

4.4.2.2 Packing material from leg and foot (left) [10] (Ha7386/856)

The results of the Py-GC/MS analysis are shown in Figure 4.5a. Present as significant components are a series of alkene/alkane doublets (C_6 to C_{21}), with the n - C_{15} homologue most abundant. In lower abundance are the *cis*- and *trans*-2-alkenes in the C_8 to C_{18} carbon number range and a number of alkadienes, including the 1,3-alkadienes (C_9 to C_{15}), a C_9 1,4-alkadiene and the 2,4-alkadienes (C_9 and C_{12} and methyl branched i - C_{10} , i - C_{11} and i - C_{15}). Methyl ketones (C_6 to C_{19}), maximising at n - C_9 were major components of the pyrolysate, along with lesser amounts of the more unusual ethyl (3-) ketones (C_{10} , C_{11} and C_{18}). Saturated ($C_{11:0}$ and $C_{19:0}$) and the $C_{19:1}$ unsaturated (2-ene) propyl (4-) ketones were present along with abundant mid-chain ketones (5- to 9-ones) in the C_{10} to C_{18} carbon number range, maximising at $C_{15:0}$ with the co-eluting 7-pentadecanone and 8-pentadecanone, these latter two comprising the major peak in the pyrogram. At later retention times (55 to 69 min), long chain diketones were observed in appreciable amounts, being identified as 4,13-octadecanedione (m/z 43, 58, 71, 85, 99, 115, 127 and $[M-71]^+$ 211), 4,12-octadecanedione (m/z 43, 58, 71, 85, 113, 129, 141 and $[M-71]^+$ 211), 4,11-octadecanedione (m/z 43, 57, 71, 85, 127, 143, 155 and $[M-71]^+$ 211), 4,11-octadec-5(?)-enedione (m/z 43, 57, 71, 85, 97, 127, 141, 155 and $[M-71]^+$ 209), 2,14-octadecanedione, 2,13-octadecanedione, 2,12-octadecanedione, 2,11-octadecanedione, 2,10-octadecanedione and 2,9-octadecanedione. Significant constituents of the pyrolysate included cyclic ketones (C_5 to C_7), alicyclic and aromatic hydrocarbons, aldehydes in the C_7 to C_9 carbon number range, and the $C_{8:0}$, $C_{16:0}$ and $C_{18:0}$ nitriles. The pyrolysate constitutes the majority (>90% w/w) of the sample (i.e. *cf.* TD), indicating the presence of abundant bound lipids and pointing to a high degree of polymerisation.

4.4.2.3 Mud packing from thoracic cavity [11] (7386/804)

The results of the Py-GC/MS analysis displayed a series of alkene/alkane doublets (C_6 to C_{16}), with the n - C_{15} homologue most abundant. Also identified were the methyl ketones (C_5 to C_{10} , C_{15} and C_{17}), maximising at n - C_{17} , along with the more unusual mid-chain ketones, 7-pentadecanone, 8-pentadecanone, 7-hexadecanone (m/z 57, 71, 85, 113, 128, 141) and 8-hexadecanone (m/z 57, 71, 99, 127, 142 and 155). Eluting at 55.2 min, a long chain diketone was observed as a significant component and identified as 2,15-octadecane-

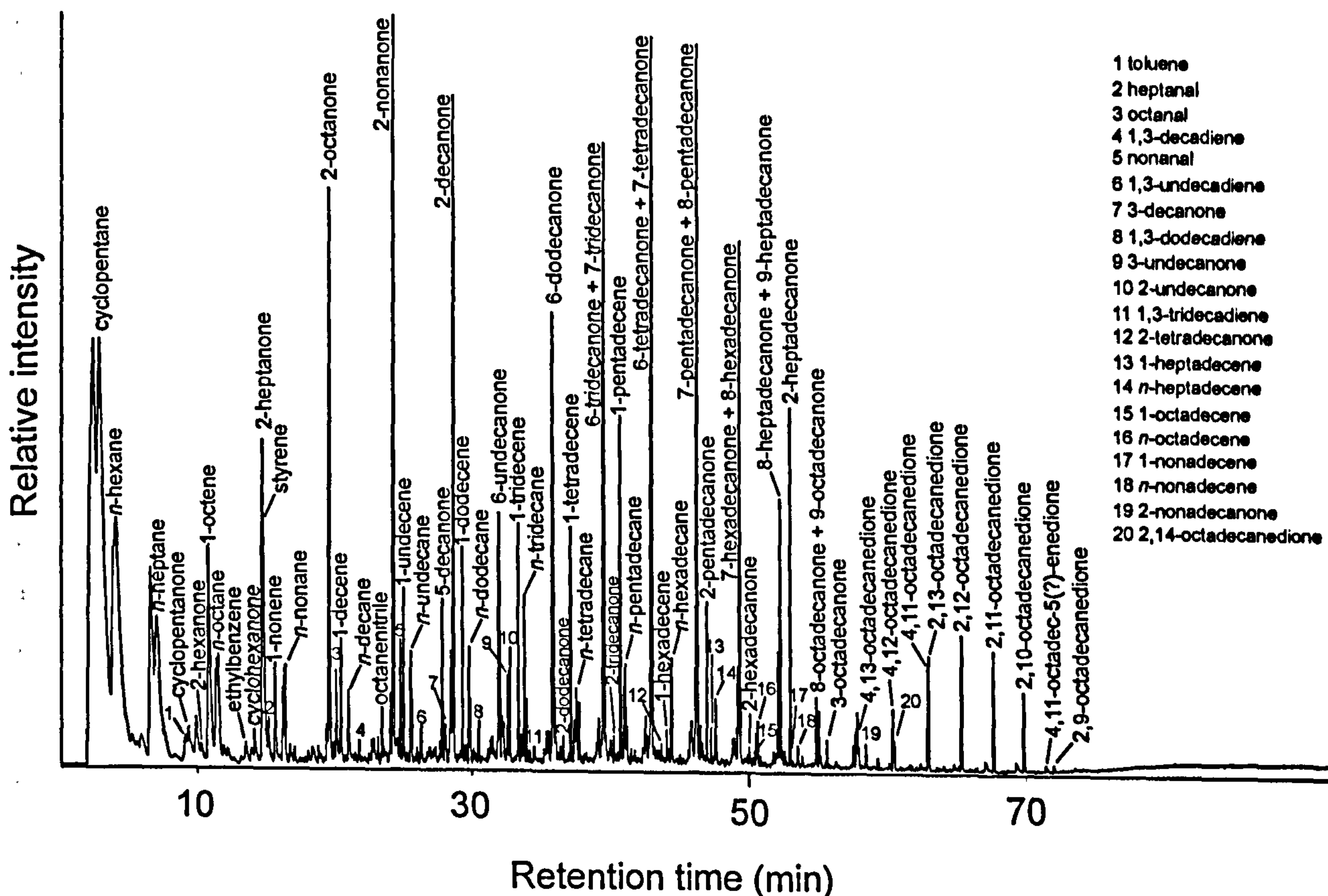


Figure 4.5a Total ion chromatogram of the pyrolysis profile (610°C/10s after TD at 310°C/10s) of packing material from the left leg and foot [10] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

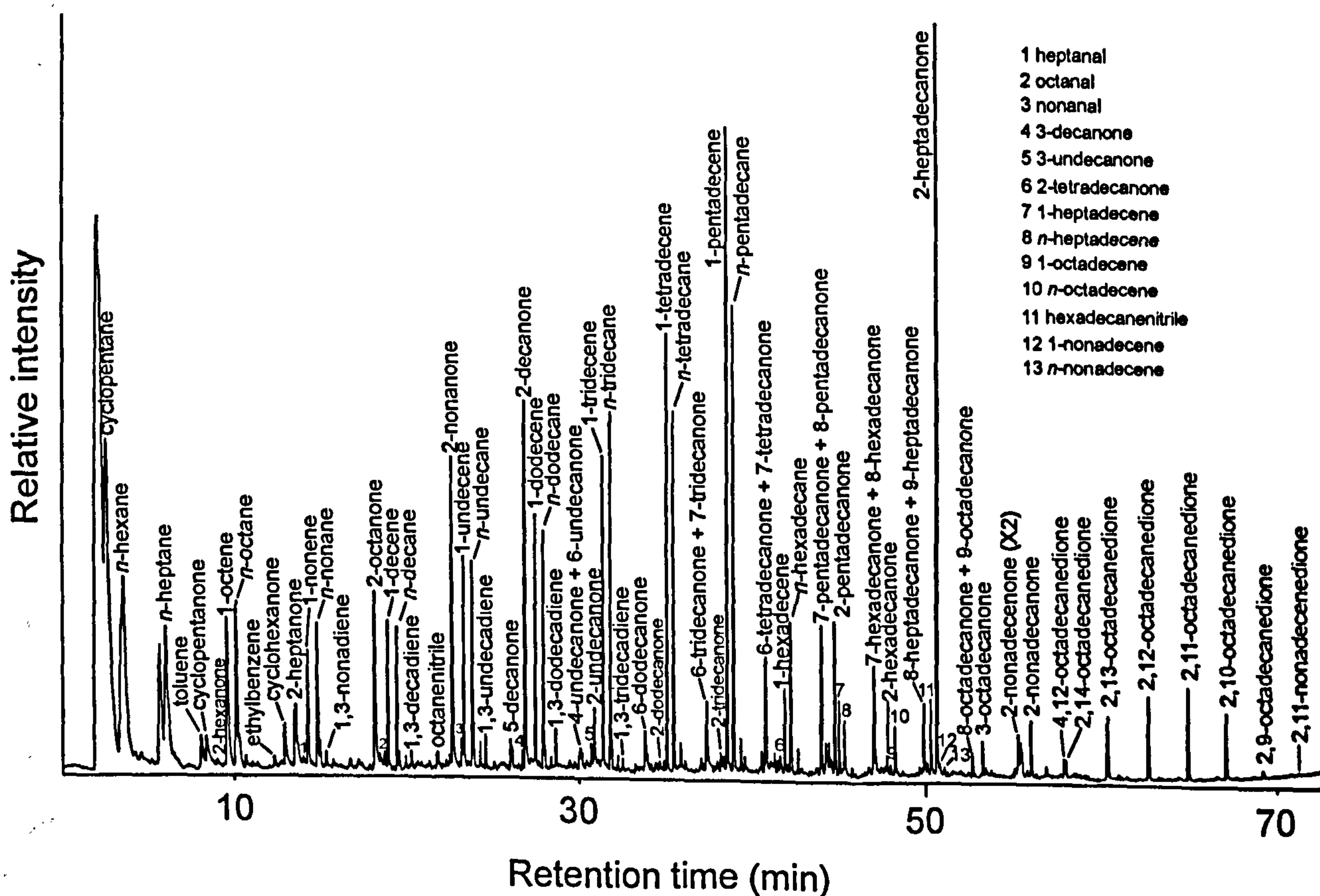


Figure 4.5b Total ion chromatogram of the pyrolysis profile (610°C/10s after TD at 310°C/10s) of packing material/wrapping from the right calf [4] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

dione (m/z 58, 71, 87, 99 and $[M-43]^+$ 239). Cyclopentane and toluene were also identified. The pyrolysate constitutes the whole (100% w/w) of the sample (i.e. *cf.* TD), indicating the presence of abundant bound lipids pointing to a high degree of polymerisation.

4.4.2.4 Packing material/wrapping from right calf [4] (7386/760)

The results of the Py-GC/MS analysis are shown in Figure 4.5b. The pyrogram is dominated by alkene/alkane doublets (C_6 to C_{21}), with the n - C_{15} homologue most abundant. In lower abundance are the *cis*- and *trans*-2-alkenes in the C_8 to C_{18} carbon number range and a number of alkadienes, including the 1,3-alkadienes (C_9 to C_{15}), a C_9 1,4-alkadiene and the 2,4-alkadienes (C_9 and C_{12} and methyl branched i - C_{10} , i - C_{11} and i - C_{15}). Methyl ketones (C_6 to C_{19}), maximising at n - C_{17} , along with the more unusual ethyl (3-) ketones (C_{10} , C_{11} and C_{18}) were also identified. The saturated ($C_{11:0}$ and $C_{19:0}$) and $C_{19:1}$ unsaturated (2-ene) propyl (4-) ketones were present along with abundant mid-chain ketones (5- to 9-ones) in the C_{10} to C_{18} carbon number range, maximising at $C_{15:0}$ with 7-pentadecanone (m/z 57, 71, 85, 113, 128, 141 and M^+ 226) and 8-pentadecanone (m/z 57, 71, 99, 127, 142, 155 and M^+ 226). At later retention times (58 to 71 min), long chain diketones were observed in appreciable amounts, being identified in order of elution as 4,12-octadecanedione, 2,14-octadecanedione, 2,13-octadecanedione, 2,12-octadecanedione, 2,11-octadecanedione, 2,10-octadecanedione, 2,9-octadecanedione and 2,11-nonadec-8(?)-enedione (m/z 57, 71, 141, 157, $[M-43]^+$ 251). Cyclic ketones (C_5 to C_7) were also present. Minor constituents of the pyrolysate included alicyclic and aromatic hydrocarbons, the latter being dominated by styrene (m/z 51, 78 and M^+ 104), aldehydes in the C_7 to C_9 carbon number range, and the $C_{8:0}$, $C_{16:0}$ and $C_{18:0}$ nitriles. The pyrolysate constitutes the majority (~90% w/w) of the sample (i.e. *cf.* TD), indicating the presence of abundant bound lipids pointing to a high degree of polymerisation.

4.4.2.5 Piece of 'resin'-soaked wrapping from left ankle/talus [1] (H7386/839)

The results of the Py-GC/MS analysis are shown in Figure 4.6a. The pyrogram was dominated by a series of alkene/alkane doublets (C_6 to C_{16}), with the n - C_8 homologue most abundant. Also identified were the methyl ketones (C_7 to C_{11} , C_{15} and C_{17}), maximising at n - C_{10} . Cyclopentane, cyclohexanone and toluene were also identified.

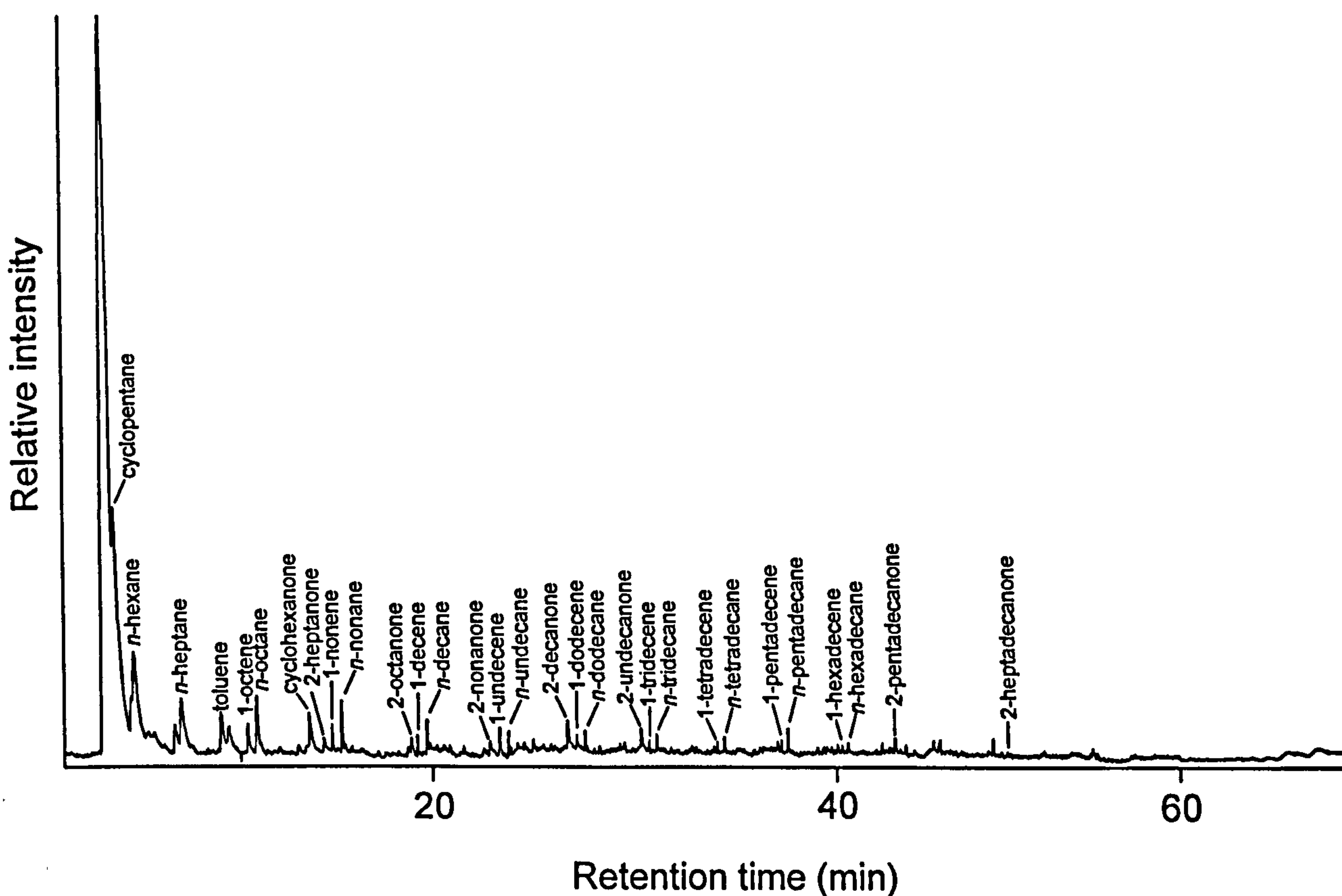


Figure 4.6a Total ion chromatogram of the pyrolysis profile (610°C/10s after TD at 310°C/10s) of a piece of 'resin'-soaked wrapping from the left ankle/talus [1] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

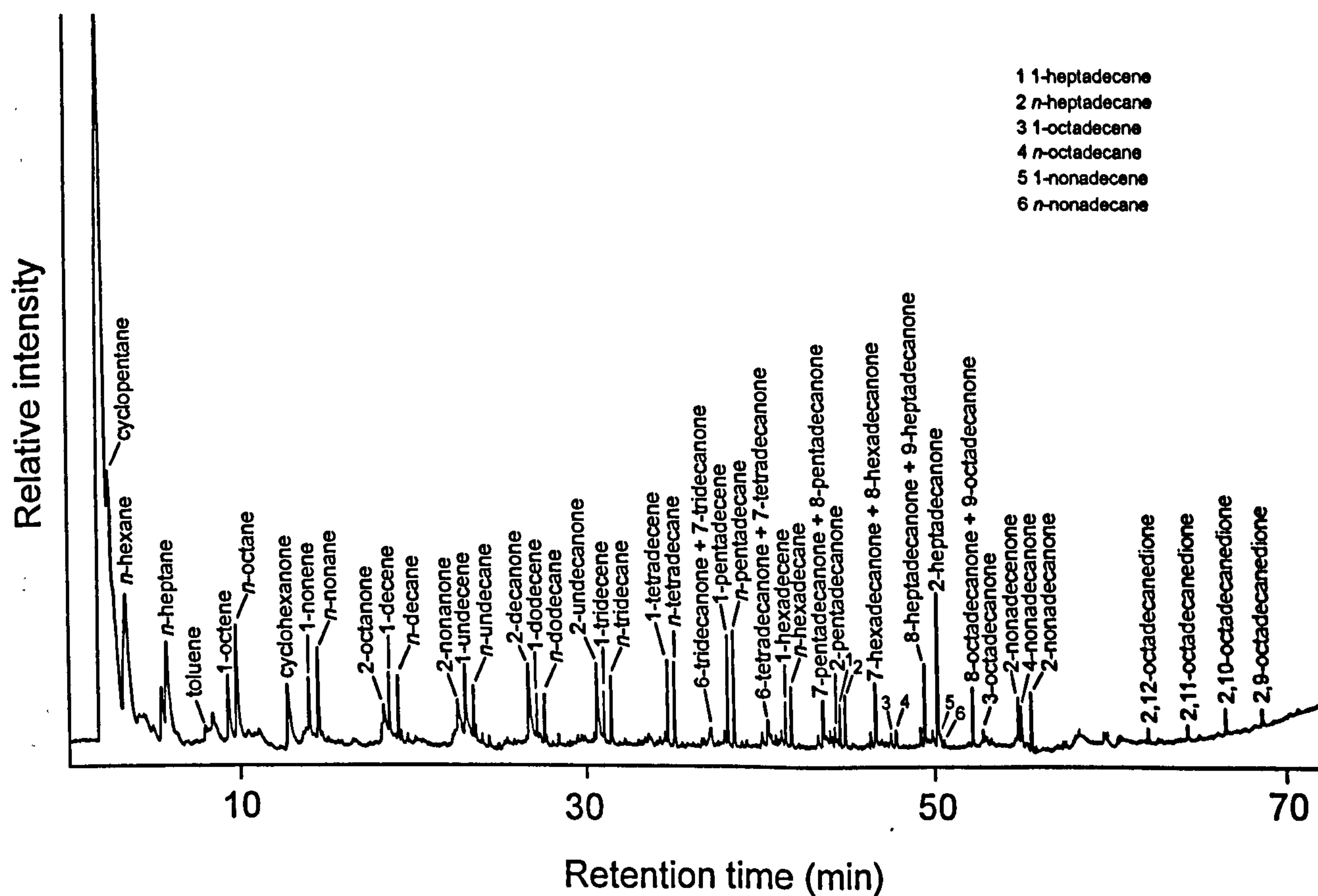


Figure 4.6b Total ion chromatogram of the pyrolysis profile (610°C/10s after TD at 310°C/10s) of wrapping/tissue from right calf [5] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

4.4.2.6 Threads from wrappings from left ankle/talus [3] (H7386/839)

The results of the Py-GC/MS analysis revealed an abundance of furan and pyran derivatives, almost certainly deriving from the cellulose which constitutes the linen threads.

4.4.2.7 Wrapping fragments [2] (Ha7386/890)

The results of the Py-GC/MS analysis displayed a series of alkene/alkane doublets (C_6 to C_{24}), with the n - C_8 homologue most abundant. Also identified were the methyl ketones (C_8 to C_{11} and C_{15}), maximising at n - C_{10} . The free fatty acids $C_{16:0}$ and $C_{18:1}$ were also observed, the abundances of these acids indicating their origin from a bound fraction of this sample, either as part of the polymeric lipid or as constituents trapped in the sample matrix. Cyclopentane, cyclohexanone and toluene were also identified.

4.4.2.8 Wrapping/tissue from right calf [5] (7386/760)

The results of the Py-GC/MS analysis are shown in Figure 4.6b. The pyrogram is dominated a series of alkene/alkane doublets (C_6 to C_{19}), with the n - C_{15} homologue most abundant. Also identified were the methyl ketones (C_8 to C_{11} , C_{15} , C_{17} and C_{19}), maximising at n - C_{17} , along with a minor amount of the $C_{18:0}$ ethyl (3-) ketone and a more appreciable amount of the $C_{19:0}$ propyl (4-) ketone. Significant quantities of mid-chain ketones (6- to 9-ones) in the C_{13} to C_{18} carbon number range, maximising at $C_{17:0}$ were also present. At later retention times (62 to 69 min), long chain diketones were observed in moderate amounts, being identified as 2,12-octadecanedione, 2,11-octadecanedione 2,10-octadecanedione and 2,9-octadecanedione. Cyclopentane, cyclohexanone and toluene were also identified.

4.4.2.9 Muscle fibres from head of right femur [7] (Ha7386/945)

The results of the Py-GC/MS analysis revealed a series of alkene/alkane doublets (C_6 to C_{17}), with the n - C_{15} homologue most abundant. Also significant were the C_{15} and C_{17} methyl ketones, although the shorter chain homologues (C_7 to C_{11}) observed in previous samples were absent. Cyclopentane, and toluene were also identified. The pyrolysate constitutes a relatively minor portion (<10% w/w) of the sample (i.e. cf. TD), indicating that the sample had not undergone a significant degree of polymerisation, consisting largely of free lipids.

4.4.2.10 'Resinous' material/muscle tissue from left hip/base of spine [8] (Ha7386/948)

The results of the Py-GC/MS analysis revealed a series of alkene/alkane doublets (C_6 to C_{18}), with the n - C_8 homologue most abundant. In lower abundance were the *cis*- and *trans*-2-alkenes in the C_8 to C_{13} carbon number range and a number of 1,3-alkadienes (C_{11} to C_{13}). Significant components included the C_9 to C_{11} methyl ketones, with relatively minor amounts of the C_{15} , C_{17} and C_{19} homologues. Other notable constituents of the pyrolysate included cyclic ketones (C_5 to C_7), alicyclic and aromatic hydrocarbons (C_6 to C_9), and the $C_{9:0}$, $C_{10:0}$, $C_{16:2}$, $C_{16:1}$, $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ nitriles. The pyrolysate constitutes a relatively minor portion (~10% w/w) of the sample (i.e. *cf.* TD), indicating that the sample had not undergone a significant degree of polymerisation, consisting largely of free lipids.

4.4.2.11 'Resinous' material from left side of upper spine [15] (Ha7386/908)

The results of the Py-GC/MS analysis are shown in Figure 4.7a. The pyrogram revealed a series of alkene/alkane doublets (C_6 to C_{16}), with the n - C_8 homologue most abundant. Also identified were the methyl ketones (C_9 to C_{11} , C_{17} and C_{19}), maximising at n - C_{10} . Other notable constituents of the pyrolysate included cyclopentanone [the major component (after CO_2) of the profile], aromatic hydrocarbons (C_6 to C_9), and the $C_{16:2}$, $C_{16:1}$, $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ nitriles. The pyrolysate constitutes a relatively significant portion (~50% w/w) of the sample (i.e. *cf.* TD), indicating that the sample had undergone a significant degree of polymerisation.

4.4.2.12 'Resin' around the mouth [12] (Ha7386/686)

The results of the Py-GC/MS analysis are shown in Figure 4.7b. The pyrogram revealed a series of alkene/alkane doublets (C_6 to C_{16}), with the n - C_8 homologue most abundant. Also identified were the methyl ketones (C_9 to C_{11} , C_{17} and C_{19}), maximising at n - C_{10} . Other notable constituents of the pyrolysate included cyclopentanone [the major component (after CO_2) of the profile], aromatic hydrocarbons (C_6 to C_9), and the $C_{16:2}$, $C_{16:1}$, $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ nitriles. The pyrolysate constitutes a relatively minor portion (~10% w/w) of the sample (i.e. *cf.* TD), indicating that the sample had not undergone a significant degree of polymerisation, consisting largely of free lipids.

4.4.2.13 Whitish 'efflorescence' from head of right femur (a) [6] (Ha7386/945)

The results of the Py-GC/MS revealed a series of alkene/alkane doublets (C_8 to C_{24}) maximising n - C_9 to C_{11} . Free fatty acids ($C_{7:0}$ to $C_{9:0}$) were also observed along with $C_{16:0}$ and $C_{18:0}$, the abundances of these acids indicating their origin from a bound fraction of

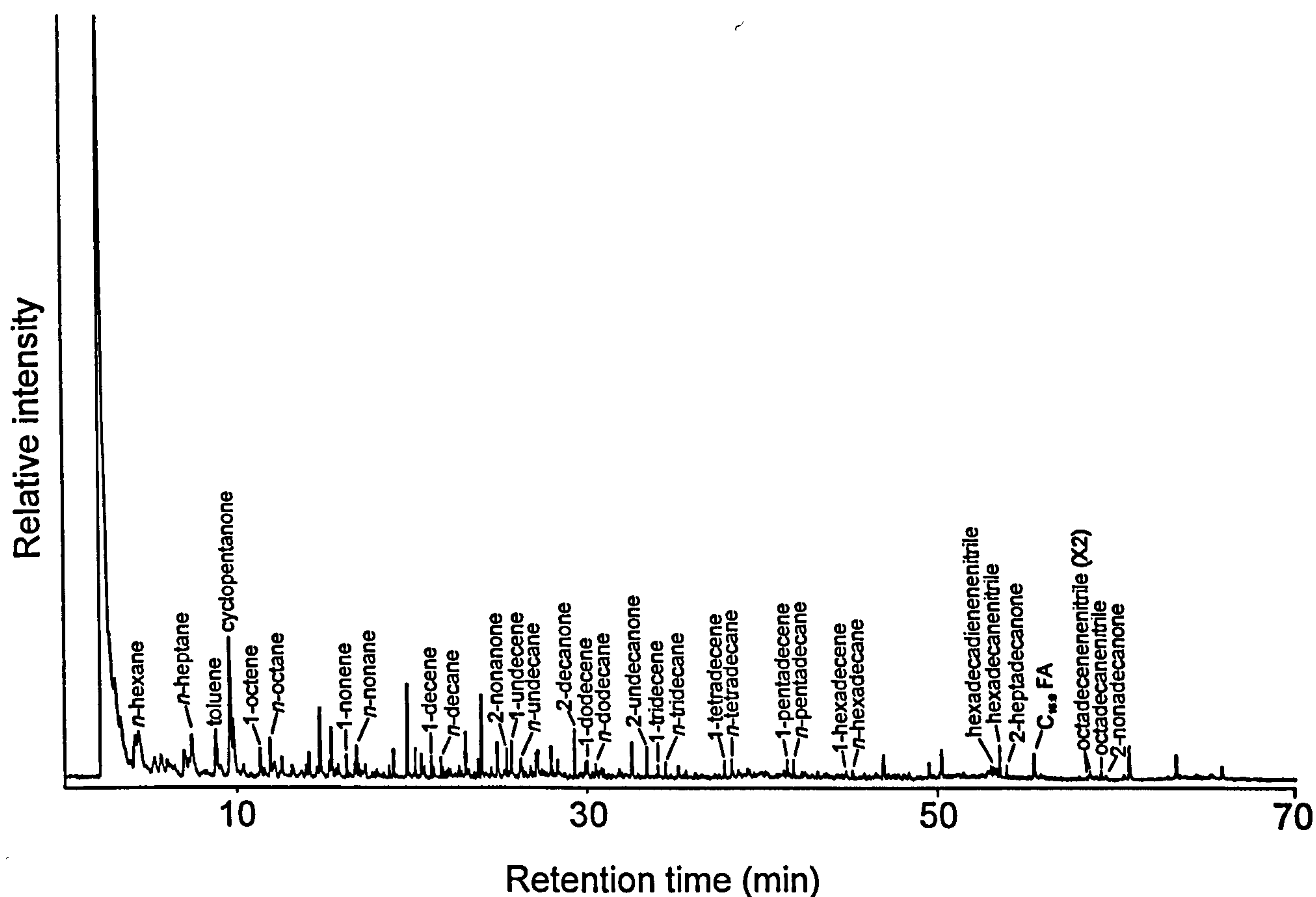


Figure 4.7a Total ion chromatogram of the pyrolysis profile (610°C/10s after TD at 310°C/10s) of 'resinous' material from the left side of the upper spine [15] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

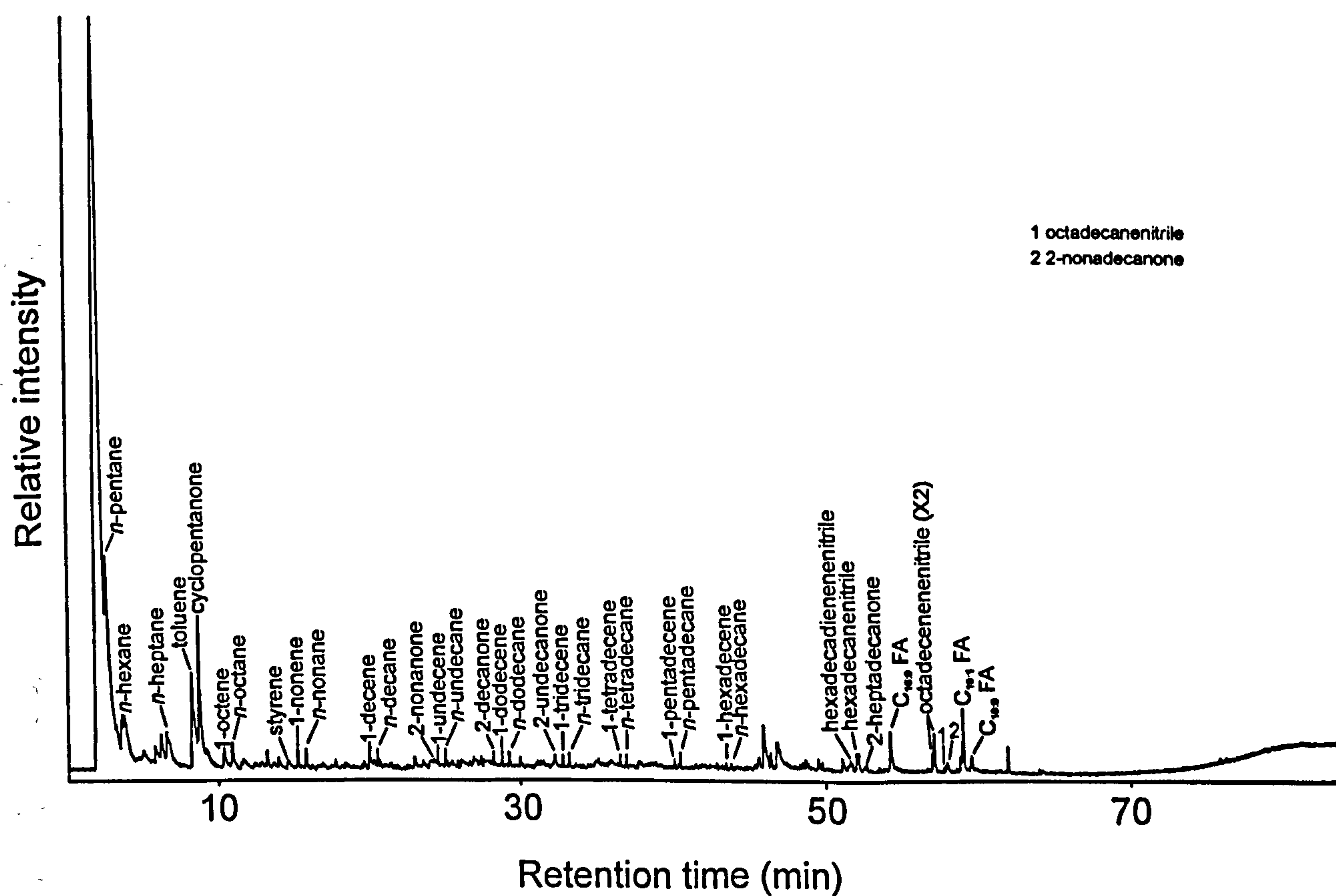


Figure 4.7b Total ion chromatogram of the pyrolysis profile (610°C/10s after TD at 310°C/10s) of 'resin' around the mouth [12] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

this sample, either as part of the polymeric lipid or as constituents trapped in the sample matrix.

4.4.14 Whitish 'efflorescence' from head of right femur (b) [17] (Ha7386/945)

The results of the Py-GC/MS analysis are shown in Figure 4.8. The pyrogram revealed a series of alkene/alkane doublets (C_8 to C_{18}) and the monocarboxylic acids $C_{16:1}$, $C_{16:0}$, $C_{18:1}$, and $C_{18:0}$.

4.4.15 Whitish 'efflorescence' from head of right femur (c) [18] (Ha7386/945)

The results of the Py-GC/MS analysis revealed a series of alkene/alkane doublets (C_8 to C_{18}) and the monocarboxylic acids $C_{16:1}$, $C_{16:0}$, $C_{18:1}$, and $C_{18:0}$ as major components.

4.4.3 GC/MS – Acid fractions (* 4.4.3.6, 4.4.3.13, 4.4.3.14 and 4.4.3.15 are TLEs)

4.4.3.1 Packing material (natron?) between wrappings and skin of left(?) upper forearm [9] (Ha7386/731)

The results for the acid fraction analysed by GC/MS revealed a series monocarboxylic acids (C_9 to C_{18}) as the major components (as their TMS derivatives), with $C_{16:0}$ (m/z 73, 117, 132, 145, $[M-15]^+$ 313 and M^+ 328) $C_{14:0}$ (m/z 73, 117, 132, 145, $[M-15]^+$ 285 and M^+ 300) and $C_{9:0}$ (m/z 73, 117, 132, 145, $[M-15]^+$ 215 and M^+ 230) predominating. The distribution of monocarboxylic acids differed markedly from that obtained using TD, presumably due to the relative volatility of the short chain fatty acids ($C_{5:0}$ to $C_{9:0}$) in comparison with the $C_{14:0}$ to $C_{18:0}$ acids which are usually the dominant constituents of degraded acyl lipids. The more volatile acids observed in the TD would be expected to be disproportionately lost during the evaporation stage of the sample preparation. The relatively high abundance of the $C_{9:0}$ fatty acid (and the presence of what were probably the $C_{6:0}$ to $C_{8:0}$ fatty acids observed in the GC analysis, too volatile to be observed in the GC/MS with the conditions utilised) suggest that they are major components in the packing material. The monounsaturated fatty acids (again as their TMS derivatives) $C_{16:1}$ (m/z 73, 117, 129 and $[M-15]^+$ 311) and $C_{18:1}$ (m/z 73, 117, 129 and $[M-15]^+$ 339) were significant components of the fraction, as were the $C_{18:1}$ 8- (characterised by m/z 73, 129, and 241), 9- (m/z 73, 129, and 227) and 10-monohydroxy (m/z 73, 129, and 329) carboxylic acids, although notably the 11-hydroxy compound was absent (see discussion). Present in similar abundance to the $C_{18:1}$ hydroxy acids were the more unusual $C_{16:1}$ hydroxy fatty acids, identified as the 8-hydroxy (m/z 73, 129 and 213) and 9-hydroxy (m/z 73, 129 and 199)

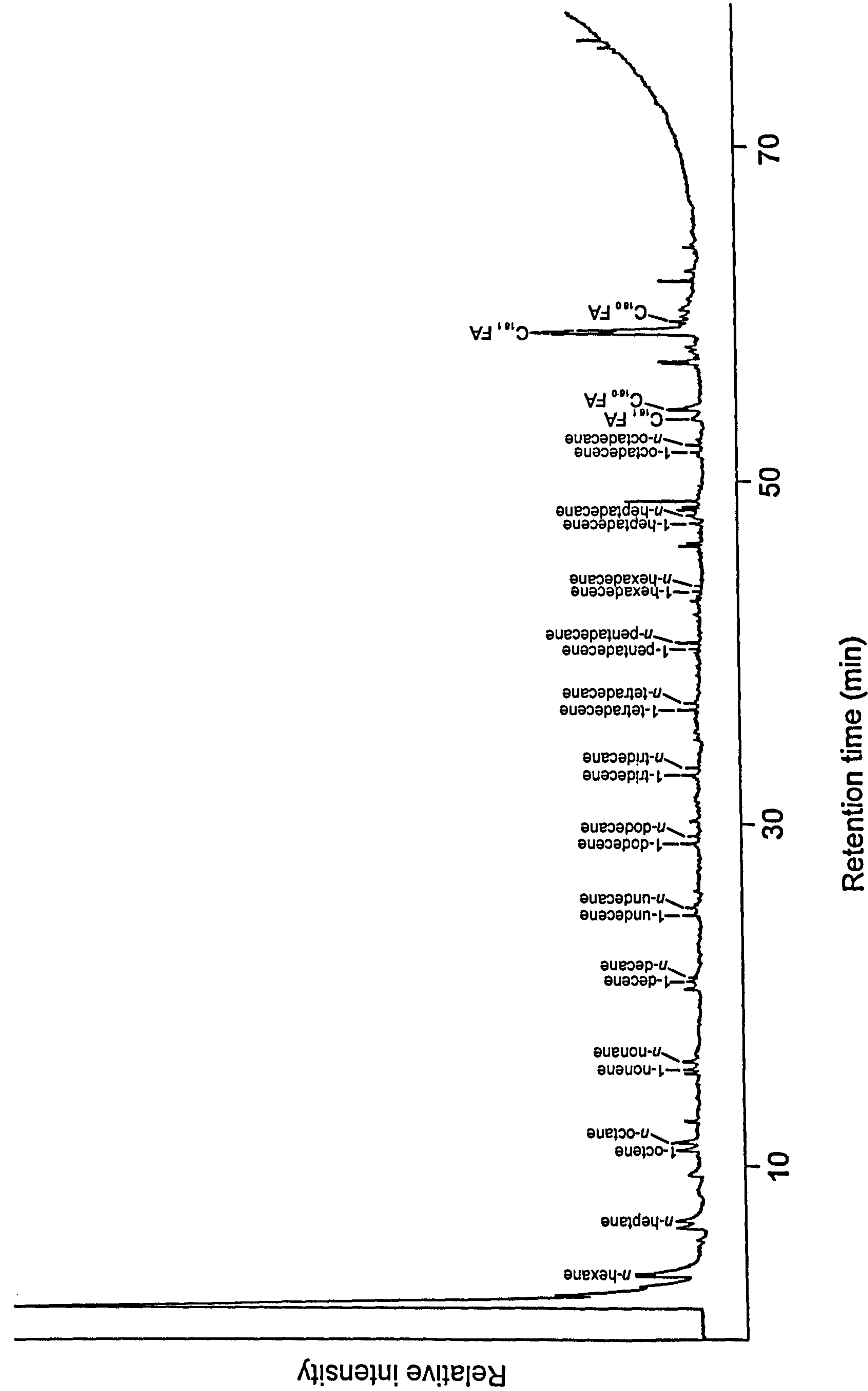


Figure 4.8 Total ion chromatogram of the pyrolysis profile (610°C/10s after TD at 310°C/10s) of 'whitish effluorescence' from the head of the right femur (b) [17] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

and 10-hydroxy (m/z 73, 129 and 329) isomers, suggesting a high proportion of $C_{16:1}$ fatty acid in the original sample, prior to burial. Short chain monohydroxy fatty acids were also detected, albeit as relatively minor components, being identified as the $C_{7:0}$ and $C_{8:0}$ ω -hydroxy acids (characterised by m/z 73, 75, 147, 185 and $[M-15]^+$ 275; and (m/z 73, 75, 147, 199 and $[M-15]^+$ 289 respectively). In moderate abundance was 9,10-dihydroxyoctadecanoic acid (as its di-TMS derivative) and characterised by m/z 73, 215 and 317, with the *threo* and *erythro* isomers present in equal abundance. The more unusual 8,10-dihydroxyoctadecanoic (m/z 73, 215 and 303) and 9,11-dihydroxyoctadecanoic (m/z 73, 201 and 317) acids were also observed. These polar hydroxy (mono- and di-) acids were not seen using TD; presumably these more highly functionalised compounds are not amenable TD-GC/MS in their underivatised form. Relatively little extractable material was obtained; this is consistent with the findings of the sequential TD/Py-GC/MS analysis.

4.4.3.2 Packing material from leg and foot (left) [10] (Ha7386/856)

The results for the acid fraction analysed by GC/MS revealed a series monocarboxylic acids (C_9 to C_{18}) as the major components (as their TMS derivatives), with $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ predominating. The distribution of monocarboxylic acids differed markedly from that obtained using TD, presumably due to the relative volatility of the short chain fatty acids ($C_{5:0}$ to $C_{9:0}$) in comparison with the $C_{14:0}$ to $C_{18:0}$ acids which are usually the dominant constituents of degraded acyl lipids. The $C_{16:1}$ monounsaturated fatty acid was identified as a significant component of the fraction, as were the $C_{18:1}$ 8-, 9- and 10-monohydroxy carboxylic acids, although notably the 11-hydroxy compound was absent (see discussion). 9,10-dihydroxyoctadecanoic acid was present in moderate abundance, although the more unusual 8,10-dihydroxyoctadecanoic and 9,11-dihydroxyoctadecanoic acids, observed in the previous sample, were not detected. These polar hydroxy (mono- and di-) acids were not seen using TD; so presumably these more highly functionalised compounds are not amenable TD-GC/MS in their underivatised form. Very little extractable material was obtained; this is consistent with the findings of the sequential TD/Py-GC/MS analysis.

4.4.3.3 Mud packing from thoracic cavity [11] (7386/804)

The results for the acid fraction analysed by GC/MS revealed a series monocarboxylic acids (C_9 to C_{18}) as the major components, with $C_{16:0}$, $C_{18:1}$, $C_{18:0}$ and $C_{14:0}$ predominating, in decreasing order of abundance. The relatively high abundance of the $C_{9:0}$ fatty acid suggests that it is a major components of the packing material. The $C_{16:1}$ monounsaturated fatty acid was identified as a significant component of the fraction, as were the $C_{18:1}$ 8-, 9-

10- and 11-monohydroxy carboxylic acids, notably the 11-hydroxy compound being present in this sample, unlike the previous two packing materials (see discussion). In similar abundance were 9,10-dihydroxyhexadecanoic acid and 9,10-dihydroxyoctadecanoic acid (the *erythro* isomer dominating in the latter), with lesser amounts of the more unusual 8,10-dihydroxyoctadecanoic and 9,11-dihydroxyoctadecanoic acids. In addition, appreciable quantities of the C_{7:0} and C_{9:0} dicarboxylic acids were also observed. These polar hydroxy (mono- and di-) and diacids are presumably too highly functionalised to be amenable to TD-GC/MS in their underivatised form. Relatively little extractable material was obtained, this is consistent with the findings of the sequential TD/Py-GC/MS analysis.

4.4.3.4 Packing material/wrapping from right calf [4] (7386/760)

The results for the acid fraction analysed by GC/MS are shown in Figure 4.9a. The chromatogram displayed a complex distribution of carboxylic acids. These included a series of monocarboxylic acids in the C₆ to C₁₈ carbon number range with the C_{16:0}, C_{8:0}, C_{9:0} and C_{14:0} the major components in decreasing order of abundance. The relative distribution of the C_{6:0} to C_{9:0} is comparable with that observed in the TD-GC/MS analysis. However, the presence of the longer chain fatty acids (C_{16:0}, C_{14:0}, etc.) which were not observed in the TD, indicates that these short chain fatty acids have been lost disproportionately during the evaporation stages of the sample preparation. The unsaturated fatty acids were present in moderate abundance, with appreciable quantities of a wide range of hydroxy fatty acids (C₇ to C₁₈). Of the short chain fatty acids (as their TMS derivatives), the C_{8:0} to C_{10:0} 3-hydroxy (characterised by *m/z* 73, 147, [M-131]⁺, 233 and [M-15]⁺), C_{8:0} to C_{11:0} 5-hydroxy (as their TMS ether, free acid derivatives *m/z* 73, 98, 173, 188 and [M-17]⁺), C_{8:0} 6-hydroxy (*m/z* 73, 75, 131 and [M-105]⁺), C_{8:1} to C_{11:1} 6-hydroxy (*m/z* 73, 147, 155, 260, 273, [M-117]⁺ and [M-15]⁺), C_{8:0} to C_{10:0} (ω-1)-hydroxy (as their TMS ether, free acid derivatives *m/z* 73, 75, 145, 161 and [M-15]⁺) and C_{7:0} and C_{8:0} ω-hydroxy (*m/z* 73, 75, 147, [M-105]⁺, [M-31]⁺ and [M-15]⁺) fatty acids were identified, along with the δ-C_{9:0}, δ-C_{14:0}, and γ- and δ-C_{16:0} lactones. For the possible origin of these relatively unusual fatty acids see discussion. Of the longer chain hydroxy acids, the abundance of the 9,10-dihydroxyhexadecanoic acid and 9,10-dihydroxyoctadecanoic acids was appreciable, with the *threo* isomer predominating in the former, and both *threo* and *erythro* isomers equally abundant in the latter. Lesser amounts of the more unusual 8,10-dihydroxyoctadecanoic (two isomers) and 9,11-dihydroxyoctadecanoic (two isomers) acids were also detected. The C_{18:1} monohydroxy- carboxylic acid was a major component,

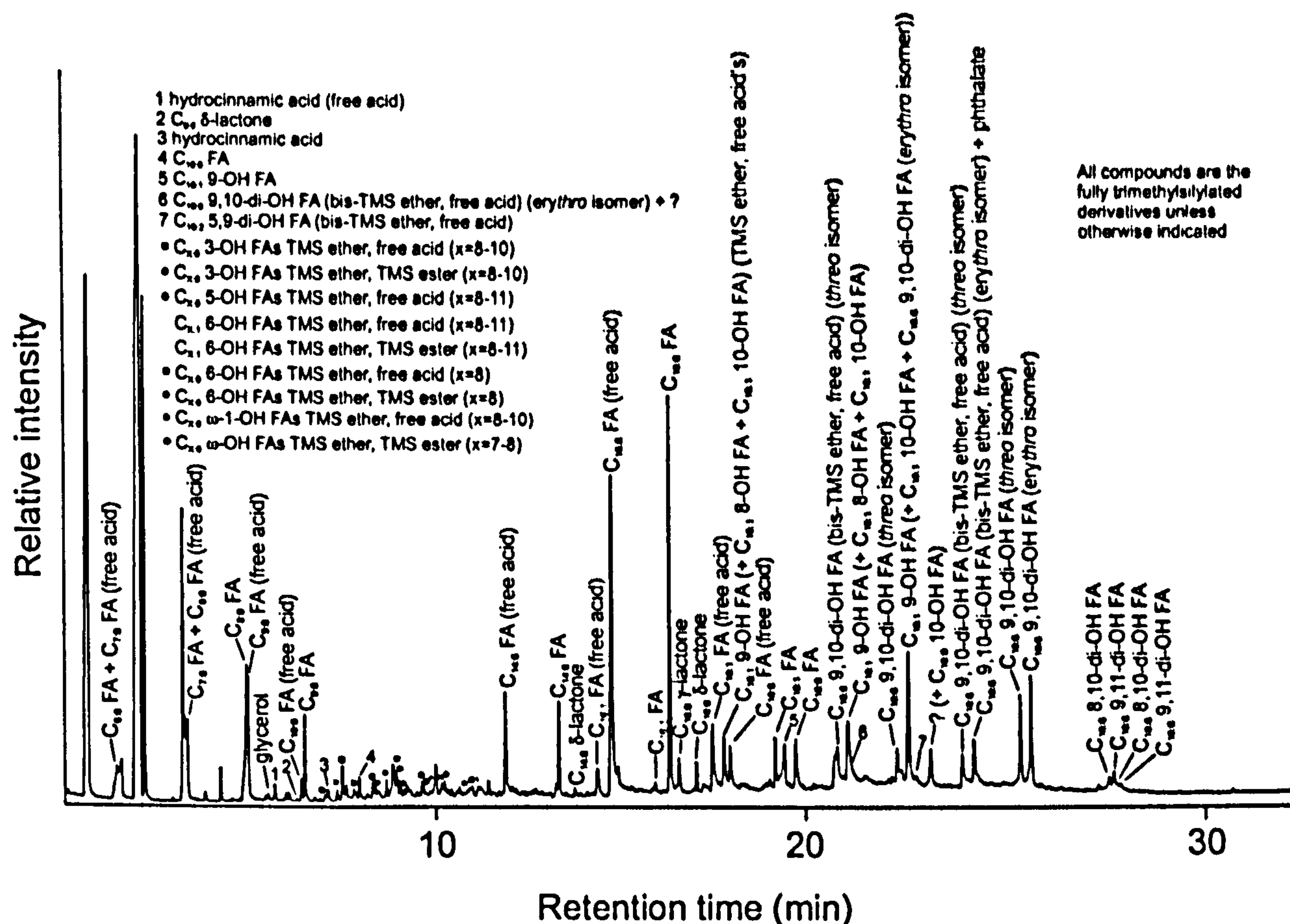


Figure 4.9a Total ion chromatogram from the GC/MS analysis of the acid fraction of packing material/ wrapping from the right calf [4] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

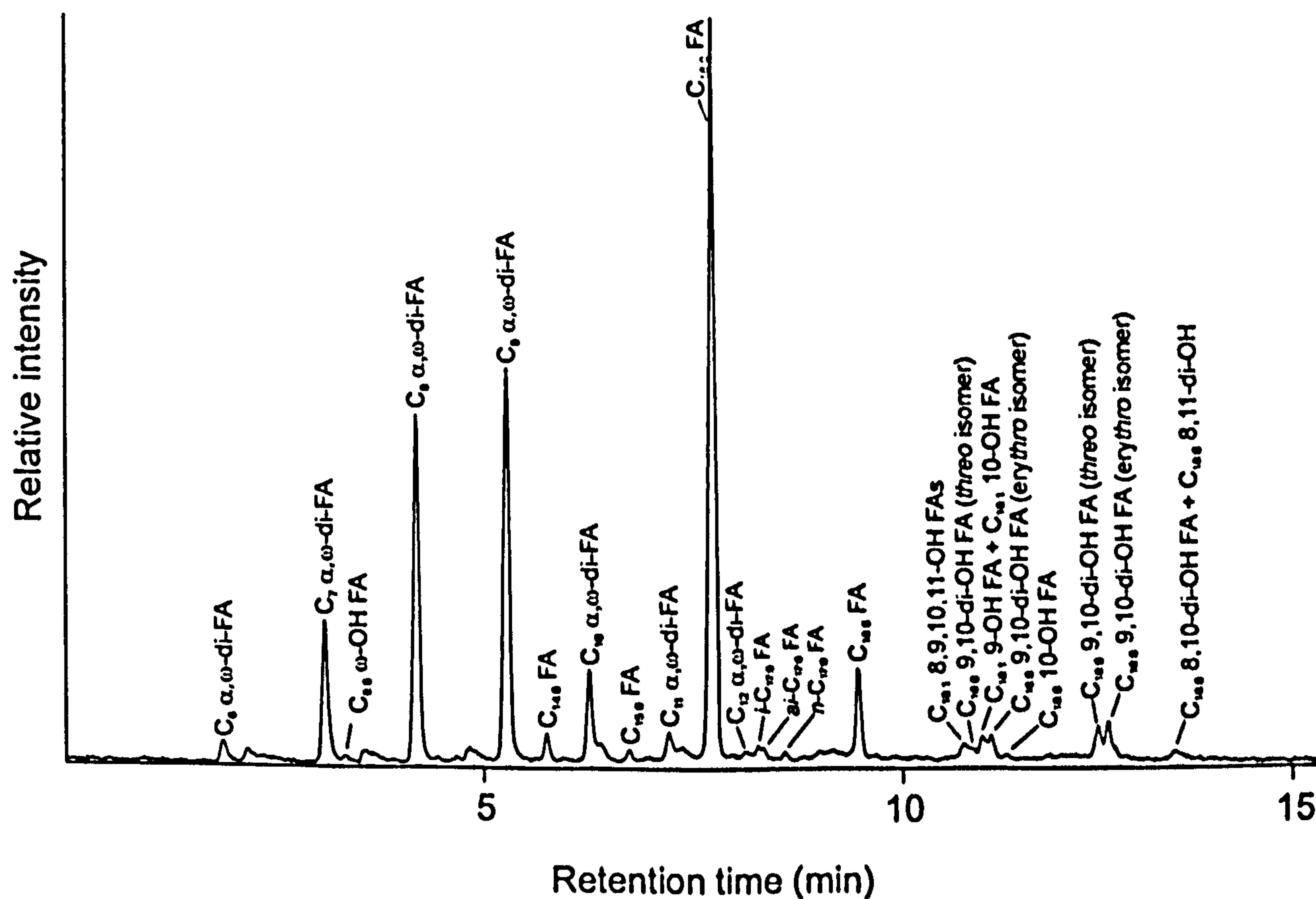


Figure 4.9b Total ion chromatogram from the GC/MS analysis of the acid fraction of a piece of 'resin'-soaked wrapping from the left ankle/talus [1] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

with the more unusual $C_{16:1}$ hydroxy acid present in appreciable abundance. Notably however, the 9-hydroxy isomer in both of these acids was predominant, with a small proportion of the 8- and 10-hydroxy compounds, with the 11-hydroxy isomer only a trace constituent. This indicates a high degree of specificity in the oxidation of the original unsaturated fatty acids likely to be present in the original fat/oil. For the possible origin of these oxidised fatty acids see discussion. Relatively little extractable material was obtained, this being consistent with the findings of the sequential TD/Py-GC/MS analysis.

4.4.3.5 Piece of 'resin'-soaked wrapping from left ankle/talus [1] (H7386/839)

The results for the acid fraction analysed by GC/MS are shown in Figure 4.9b. A series monocarboxylic acids (C_{14} to C_{18}) were observed, with $C_{16:0}$ predominating. Palmitic acid was indeed identified as the major component of the acid fraction, the $C_{14:0}$ and $C_{18:0}$ fatty acids being relatively minor components, with the distribution of monocarboxylic acids similar to that obtained using TD (see Fig. 4.2a). Minor amounts of the $C_{15:0}$ and $C_{17:0}$ branched chain fatty acids were also observed. The monounsaturated fatty acids present in the previous four samples were not identified, yet the C_6 to C_{12} dicarboxylic acids were present as significant components, C_7 , C_8 and C_9 being the major constituents of the chromatogram after $C_{16:0}$. The $C_{18:1}$ 8-, 9-, 10- and 11-monohydroxy carboxylic acids (see discussion) were identified, as was the short chain hydroxy fatty acid $C_{8:0}$ ω -OH. In similar abundance were 9,10-dihydroxyhexadecanoic acid and 9,10-dihydroxyoctadecanoic acid, the *erythro* isomers predominating in both acids, with lesser amounts of the more unusual 8,10-dihydroxyoctadecanoic and 8,11-dihydroxyoctadecanoic (m/z 73, 129, 201 and 303) acids.

4.4.3.6 Threads from wrappings from left ankle/talus [3] (H7386/839)*

The results for the total lipid extract analysed by GC/MS revealed a complex suite of lipids, the major components being a series of monocarboxylic acids (C_9 to C_{28}), with $C_{16:0}$, $C_{18:0}$ and $C_{14:0}$ predominating in decreasing order of abundance. The distribution of monocarboxylic acids was similar to that obtained using TD, although the shorter chain fatty acids were less abundant, no doubt due to the greater volatility of their TMS derivatives, preferentially lost during sample preparation. Branched chain fatty acids ($C_{15:0}$ and $C_{17:0}$) and monounsaturated fatty acids ($C_{16:1}$ and $C_{18:1}$) were significant components of the fraction, whilst present in more appreciable abundance were a series of α,ω -dicarboxylic acids (C_4 to C_{11}), with C_7 and C_9 predominating. Observed in moderate amounts were the mono- and dihydroxy carboxylic acids, with the $C_{18:1}$ monohydroxy

acids (8-, 9-, 10- and 11- isomers) present in similar amounts to the C_{18:0} 9,10-dihydroxy fatty acids, the *threo* and *erythro* isomers of similar abundance in the latter. The short chain C_{7:0} and C_{8:0} ω -hydroxy fatty acids observed in 4.4.3.1 and 4.4.3.5 were detected as minor components, along with a comparable amount of 5-hydroxyheptanoic acid (as its TMS ether, free acid and characterised by *m/z* 73, 98, 173 and 188 and [M-17]⁺ 243) and an appreciable quantity of 3,4-dihydroxybutyric acid (as its di-TMS ether, TMS ester, *m/z* 73, 147, 189, 233, 246 and [M-15]⁺ 321). Also observed were the C_{22:0} to C_{28:0} long chain fatty acids, with C_{24:0} predominating, along with lesser amounts of the C₂₇ and C₂₉ *n*-alkanes and a wax ester (the *n*-alkanes were also detected as minor components in the TD-GC/MS analysis). The neutral components were relatively minor constituents of the extractable material which was dominated by carboxylic acids.

4.4.3.7 Wrapping fragments [2] (Ha7386/890)

The results for the acid fraction analysed by GC/MS revealed a range of carboxylic acids, the chromatogram being dominated by the C_{16:0} monocarboxylic acid. The acids included saturated straight chain acids in the C₁₄ to C₁₈ carbon number range, the C_{17:0} saturated *iso*- and *anteiso*-methyl branched acids and the C_{18:1} unsaturated acid. The profile of the monocarboxylic acids, dominated by the C_{16:0} component, was comparable with the TD profile obtained for this sample. A series of α,ω -dicarboxylic acids in the C₆ to C₁₀ carbon number range were present as significant components together with comparable amounts of monohydroxy and dihydroxy carboxylic acids. Of these latter constituents, the C_{18:1} 8-, 9- and 10-monohydroxy carboxylic acids (see discussion) were identified, although notably the 11-hydroxy isomer was absent. No short chain hydroxy fatty acids were observed. In similar abundance were 9,10-dihydroxyhexadecanoic acid and 9,10-dihydroxyoctadecanoic acid, the *threo* isomers predominating in the latter, with lesser amounts of the 8,10-dihydroxyoctadecanoic and 9,11-dihydroxyoctadecanoic acids.

4.4.3.8 Wrapping/tissue from right calf [5] (7386/760)

The results for the acid fraction analysed by GC/MS are shown in Figure 4.10a. The chromatogram displayed a range of carboxylic acids, the dominant component being the C_{16:0} monocarboxylic acid. The distribution of monocarboxylic acids, dominated by palmitic acid, was similar to that obtained using TD (see Fig. 4.2b). The acids included saturated straight chain acids in the C₁₄ to C₂₂ carbon number range, the C_{17:0} saturated *iso*- and *anteiso*-methyl branched acids and the C_{18:1} unsaturated acid. A series of α,ω -dicarboxylic acids in the C₇ to C₉ carbon number range were present as significant

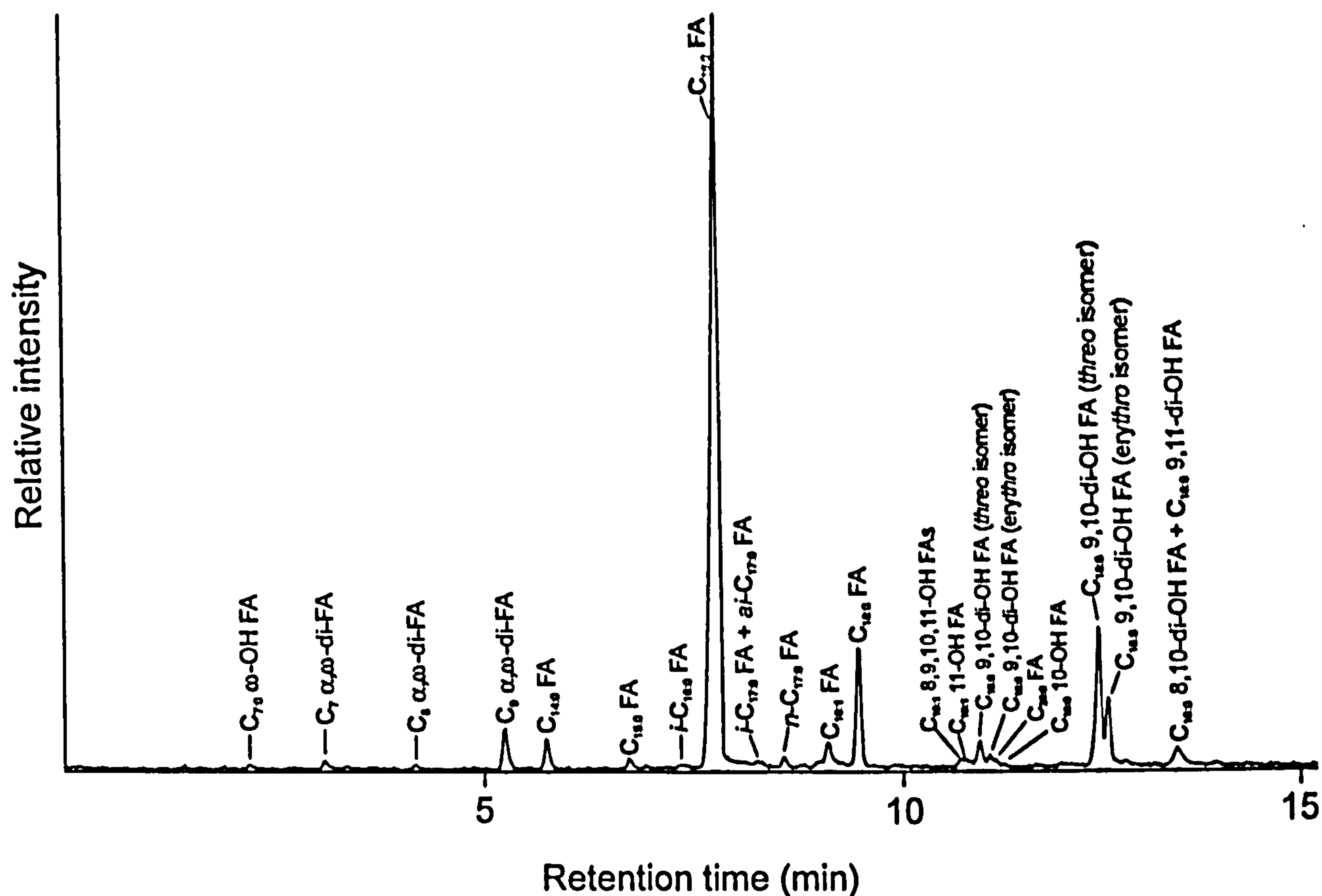


Figure 4.10a Total ion chromatogram from the GC/MS analysis of the acid fraction of wrapping/tissue from the right calf [5] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

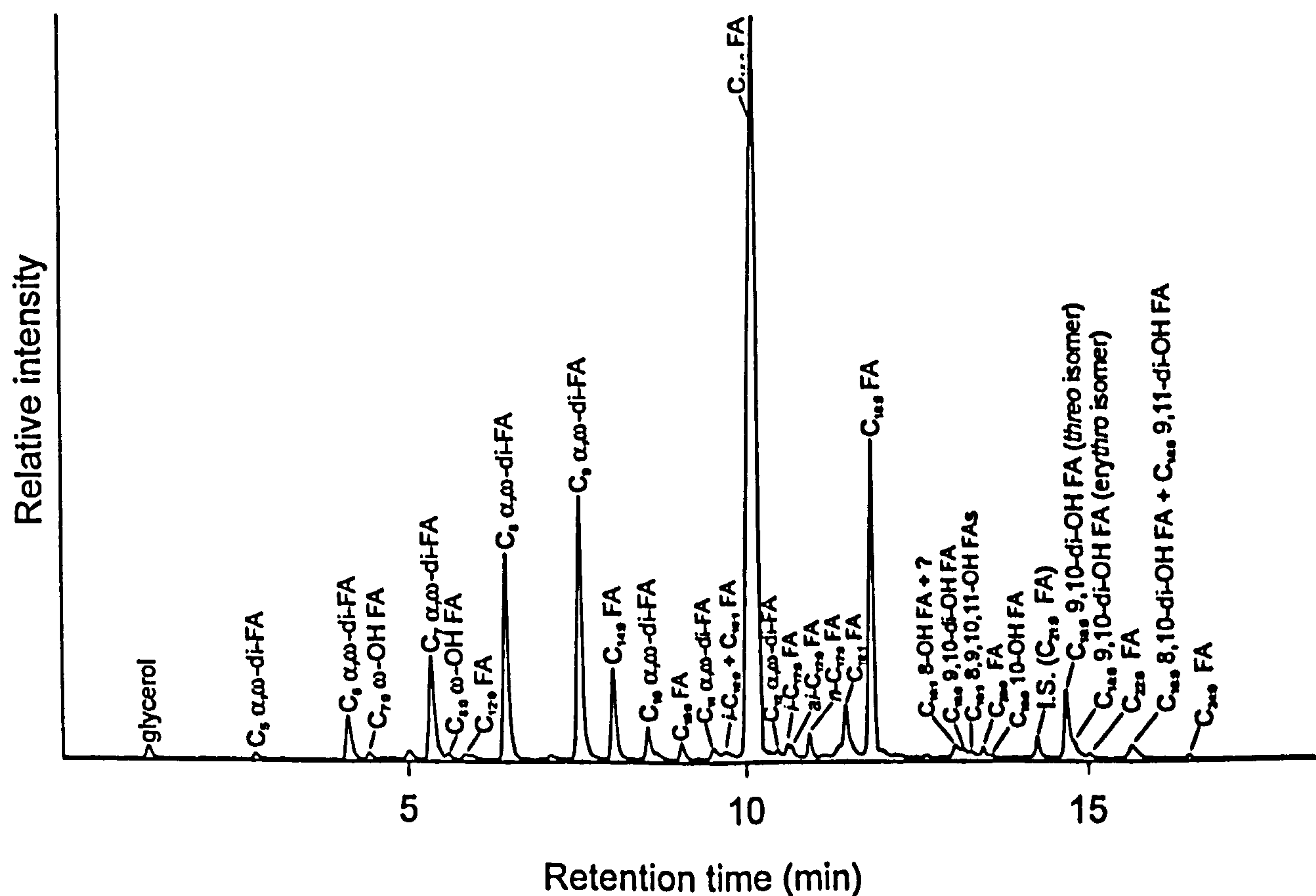


Figure 4.10b Total ion chromatogram from the GC/MS analysis of the acid fraction of 'resinous' material from the left side of the upper spine [15] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

components together with appreciable amounts of monohydroxy and dihydroxy carboxylic acids. Of these latter constituents, the C_{18:1} 8-, 9-, 10- and 11-monohydroxy carboxylic acids were identified, as was the short chain C_{7:0} ω -hydroxy fatty acid, albeit as a trace component. In higher abundance were 9,10-dihydroxyhexadecanoic acid and 9,10-dihydroxy-octadecanoic acid, the *threo* isomers predominating in both compounds, with lesser amounts of the 8,10-dihydroxyoctadecanoic and 9,11-dihydroxyoctadecanoic acids.

4.4.3.9 Muscle fibres from head of right femur [7] (Ha7386/945)

The results for the acid fraction analysed by GC/MS revealed a range of carboxylic acids, the chromatogram being dominated by a series of monocarboxylic acids (C₁₄ to C₂₀), with C_{16:0}, C_{18:1} and C_{18:0} predominating in decreasing order of abundance. The distribution of the monocarboxylic acids, dominated by the C_{16:0}, C_{18:0}, C_{18:1} and C_{14:0} components, was comparable with the TD profile obtained for this sample. Significant quantities of α,ω -dicarboxylic acids (C₄ to C₁₁), with C₈ and C₉ predominating were also observed, with lesser amounts of the C_{16:1} and branched chain fatty acids (C_{15:0} and C_{17:0}). The mono- and dihydroxy fatty acids were present in moderate abundance, with the C_{18:1} monohydroxy acids (8-, 9-, 10- and 11- isomers) present in similar amounts to the C_{16:0} and C_{18:0} 9,10-dihydroxy fatty acids (the *threo* and *erythro* isomers of similar abundance in the latter), with the more unusual 11,12-dihydroxyoctadecanoic acid (*m/z* 73, 129, 147, 187, and 345) detected as a minor component (see discussion). The aromatic acids 4-hydroxybenzoic (as its TMS ether, TMS ester, *m/z* 73, 193, 223, 267 and M⁺ 282) and 4-hydroxyhydrocinnamic (as its TMS derivative, *m/z* 73, 179, 192, 295 and M⁺ 310) were also identified as minor components of the acid fraction.

4.4.3.10 'Resinous' material/muscle tissue from left hip/base of spine [8] (Ha7386/948)

The results for the acid fraction analysed by GC/MS revealed a range of carboxylic acids, the chromatogram being dominated by a series of monocarboxylic acids (C₉ to C₂₂), with C_{16:0}, C_{18:1} and C_{18:0} predominating in decreasing order of abundance. The distribution of the monocarboxylic acids, dominated by the C_{16:0}, C_{18:0}, C_{18:1} and C_{14:0} components, was comparable with the TD profile obtained for this sample. Also observed were significant quantities of α,ω -dicarboxylic acids (C₆ to C₁₂), with C₈ and C₉ predominating, and lesser amounts of the C_{16:1} and branched chain fatty acids (C_{15:0} and C_{17:0}). The mono- and dihydroxy fatty acids were present in appreciable abundance, with the C_{18:1} monohydroxy acids (8-, 9-, 10- and 11- isomers) and C_{16:0} 9,10-dihydroxy acid being less prevalent than the C_{18:0} 9,10-dihydroxy fatty acid, the *threo* isomer being predominant in the latter. The

more unusual dihydroxy fatty acid, 11,12-dihydroxyoctadecanoic acid, was detected as a minor component. This sample was notably similar in chemical composition to the previous sample.

4.4.3.11 'Resinous' material from left side of upper spine [15] (Ha7386/908)

The results for the acid fraction analysed by GC/MS are shown in Figure 4.10b. A range of carboxylic acids was observed, the chromatogram being dominated by a series of monocarboxylic acids (C_{12} to C_{24}), with $C_{16:0}$ and $C_{18:0}$ predominating in decreasing order of abundance. The distribution of the monocarboxylic acids was comparable with the TD profile obtained for this sample (see Fig. 4.3a). Minor amounts of the $C_{16:1}$ and $C_{18:1}$, monounsaturated and $C_{17:0}$ branched chain fatty acids were identified, as were a series of α,ω -dicarboxylic acids in the C_5 to C_{12} carbon number range, with C_8 and C_9 predominating. These diacids were major constituents of the fraction and indeed the sample. Lesser amounts of monohydroxy and dihydroxy carboxylic acids were also observed, with the $C_{18:1}$ 8-, 9- 10- and 11- monohydroxy carboxylic acids and the short chain $C_{7:0}$ and $C_{8:0}$ ω -hydroxy fatty acids, and 9,10-dihydroxyhexadecanoic acid detected as minor components. In moderate abundance was the 9,10-dihydroxyoctadecanoic acid, the *threo* isomer predominating, with lesser amounts of the 8,10-dihydroxyoctadecanoic and 9,11-dihydroxyoctadecanoic acids.

4.4.3.12 'Resin' around the mouth [12] (Ha7386/686)

The results for the acid fraction analysed by GC/MS are shown in Figure 4.11a. A range of carboxylic acids was observed, the chromatogram being dominated by a series of monocarboxylic acids (C_{14} to C_{24}), with $C_{16:0}$ and $C_{18:0}$ predominating in decreasing order of abundance. The distribution of the monocarboxylic acids, was comparable with the TD profile obtained for this sample (see Fig. 4.3b). Minor amounts of the $C_{16:1}$ and $C_{18:1}$, monounsaturated and $C_{17:0}$ branched chain fatty acids were identified, as were a series of α,ω -dicarboxylic acids in the C_7 to C_{11} carbon number range, with C_8 and C_9 predominating. These diacids were significant constituents of the fraction. Lesser amounts of monohydroxy and dihydroxy carboxylic acids were also observed, with the $C_{18:1}$ 8-, 9- 10- and 11- monohydroxy carboxylic acids detected as minor components. No short chain hydroxy acids were observed. In moderate abundance was the 9,10-dihydroxyoctadecanoic acid, the *threo* isomer predominating.

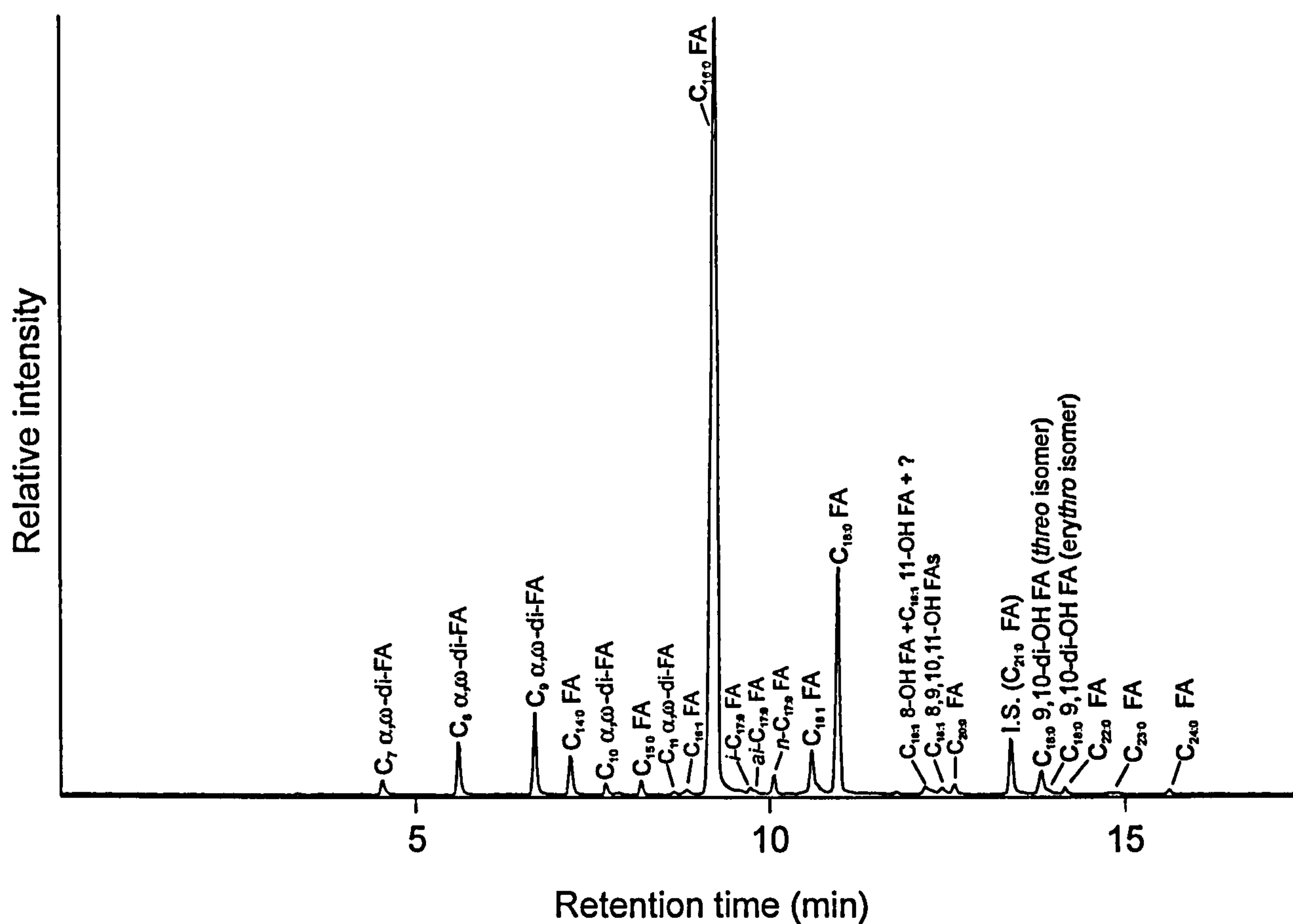


Figure 4.11a Total ion chromatogram from the GC/MS analysis of the acid fraction of ‘resin’ around the mouth [12] of the Theban priest ‘Horemkenesi’, XXIst dynasty (c. 1069-945 B.C.).

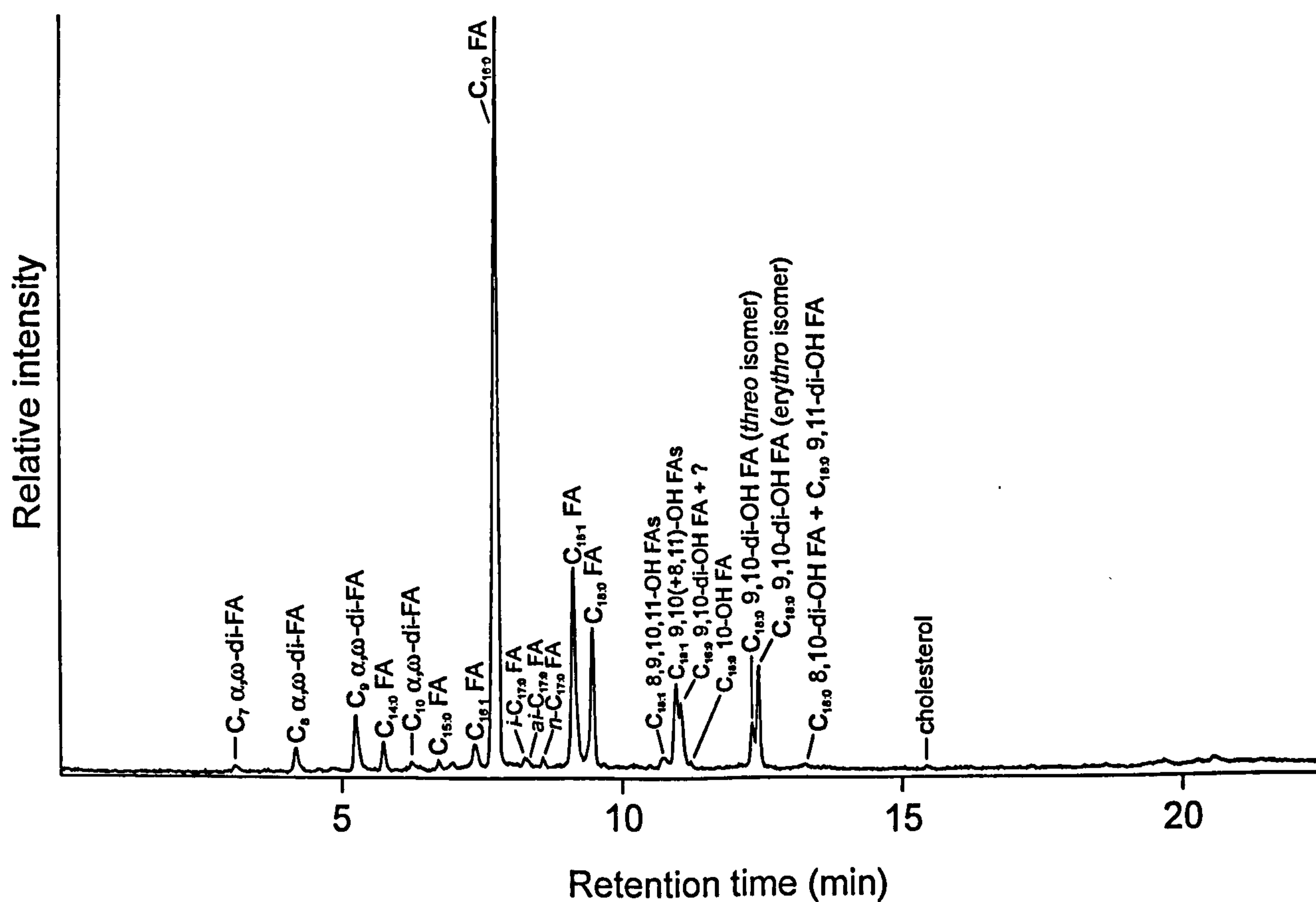


Figure 4.11b Total ion chromatogram from the GC/MS analysis of the acid fraction of ‘whitish effluorescence’ from the head of the right femur (a) [6] of the Theban priest ‘Horemkenesi’, XXIst dynasty (c. 1069-945 B.C.).

4.4.3.13 Whitish 'efflorescence' from head of right femur (a) [6] (Ha7386/945)*

The results for the total lipid extract analysed by GC/MS are shown in Figure 4.11b. A series of monocarboxylic acids (C_{14} to C_{18}) with $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ were the major components in decreasing order of abundance. The distribution of the monocarboxylic acids, was comparable with the TD profile obtained for this sample. Also present in appreciable quantities were α,ω -dicarboxylic acids (C_7 to C_{10}) with C_9 predominating. Greater amounts of monohydroxy and dihydroxy carboxylic acids were however observed, with the $C_{18:1}$ 8-, 9- 10- and 11- monohydroxy carboxylic acids, and the $C_{16:0}$ and $C_{18:0}$ 9,10-dihydroxy fatty acids in similar abundance, the *erythro* isomer predominating in the latter compound. Minor components included the $C_{16:1}$ monounsaturated and $C_{17:0}$ branched chain fatty acids, the 8,10-dihydroxyoctadecanoic and 9,11-dihydroxyoctadecanoic acids and cholesterol (TMS ether m/z 73, 129, 329, 353, 368 and M^+ 458). Cholesterol was the only neutral component detected in the solvent soluble extract.

4.4.3.14 Whitish 'efflorescence' from head of right femur (b) [17] (Ha7386/945)*

The results for the total lipid extract analysed by GC/MS revealed the $C_{18:1}$, $C_{16:0}$, $C_{18:0}$ and $C_{16:1}$ monocarboxylic acids, as the major components in decreasing order of abundance. The distribution of these monocarboxylic acids, was comparable with the TD profile obtained for this sample. Dipalmitin ($C_{16:0}/C_{16:0}$ -diacylglycerol) and monostearin ($C_{18:0}$ monoacylglycerol) were the only other components observed (excluding two phthalate contaminants), these would be too involatile in their free form to be amenable to TD-GC/MS. Very little solvent extractable material was present in this sample, indicating that this sample is of a largely inorganic nature.

4.4.3.15 Whitish 'efflorescence' from head of right femur (c) [18] (Ha7386/945)*

The results for the total lipid extract analysed by GC/MS revealed a series of monocarboxylic acids (C_{14} to C_{18}) with $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ the major components in decreasing order of abundance. The distribution of the monocarboxylic acids, was comparable with the TD profile obtained for this sample. The α,ω -dicarboxylic acids observed in the previous sample were not detected. The monohydroxy and dihydroxy carboxylic acids however, were observed, with the $C_{18:1}$ 8-, 9- 10- and 11- monohydroxy carboxylic acids, and $C_{18:0}$ 9,10-dihydroxy fatty acids present in moderate abundance. Relatively little solvent extractable material was present, suggesting that this sample is of a largely inorganic nature.

4.4.4 GC/MS –Neutral fractions

4.4.4.1 Packing material (natron?) between wrappings and skin of left(?) upper forearm [9] (Ha7386/731)

The analysis of the neutral fraction by GC/MS, revealed no detectable neutral components, confirming that the solvent extractable portion of the sample consists essentially of free fatty acids.

4.4.4.2 Packing material from leg and foot (left) [10] (Ha7386/856)

The analysis of the neutral fraction by GC/MS, revealed no detectable neutral components, confirming that the solvent extractable portion of the sample consists essentially of free fatty acids.

4.4.4.3 Mud packing from thoracic cavity [11] (7386/804)

The results for the neutral fraction analysed by GC/MS revealed that very little solvent extractable neutral components were present. A series of *n*-alkanes (C₂₅ to C₂₉), with an odd-over-even preference, were the only neutral compounds detected.

4.4.4.4 Packing material/wrapping from right calf [4] (7386/760)

The results for the neutral fraction analysed by GC/MS are shown in Figure 4.12a. The chromatogram was dominated by a series of *n*-alkanes (C₂₅ to C₃₃) with an odd-over-even preference, together with appreciable amounts of wax esters in the C₃₂ to C₅₈ carbon number range, C₄₆ and C₄₈ predominating. The C₃₂ to C₅₀ wax esters (even carbon numbers) contained predominantly the C_{16:0} acyl group, although others, particularly the C_{18:0}, were present. In contrast, the C₅₂ to C₅₈ wax esters (even carbon numbers) were found to contain predominantly the C_{20:0} acyl group, with significant amounts of the C_{22:0} to C_{26:0} acyl groups (even carbon numbers), increasing in chain length with an increase in wax ester carbon number. Also present were a series of C₃₄ to C₄₀ wax esters (even carbon numbers) with exclusively the C_{18:1} acyl moiety. A series of branched chain alkanes (C₂₆ to C₃₄), maximising at C₃₂, with an even-over-odd preference were also significant components. These branched chain alkanes contain one methyl branch mid-chain, between the 9- and the 17- positions, with the odd numbered positions predominant. Of the glycerol derivatives identified, the C_{16:0} and C_{18:0} 1-*O*-monoalkylglyceryl ethers (as their bis-TMS ethers) were major components of the neutral fraction, with moderate amounts of the C_{16:0} and C_{18:0} 1-monoacyl- and C_{18:0} 2-monoacylglycerols, and lesser quantities of the C₃₆

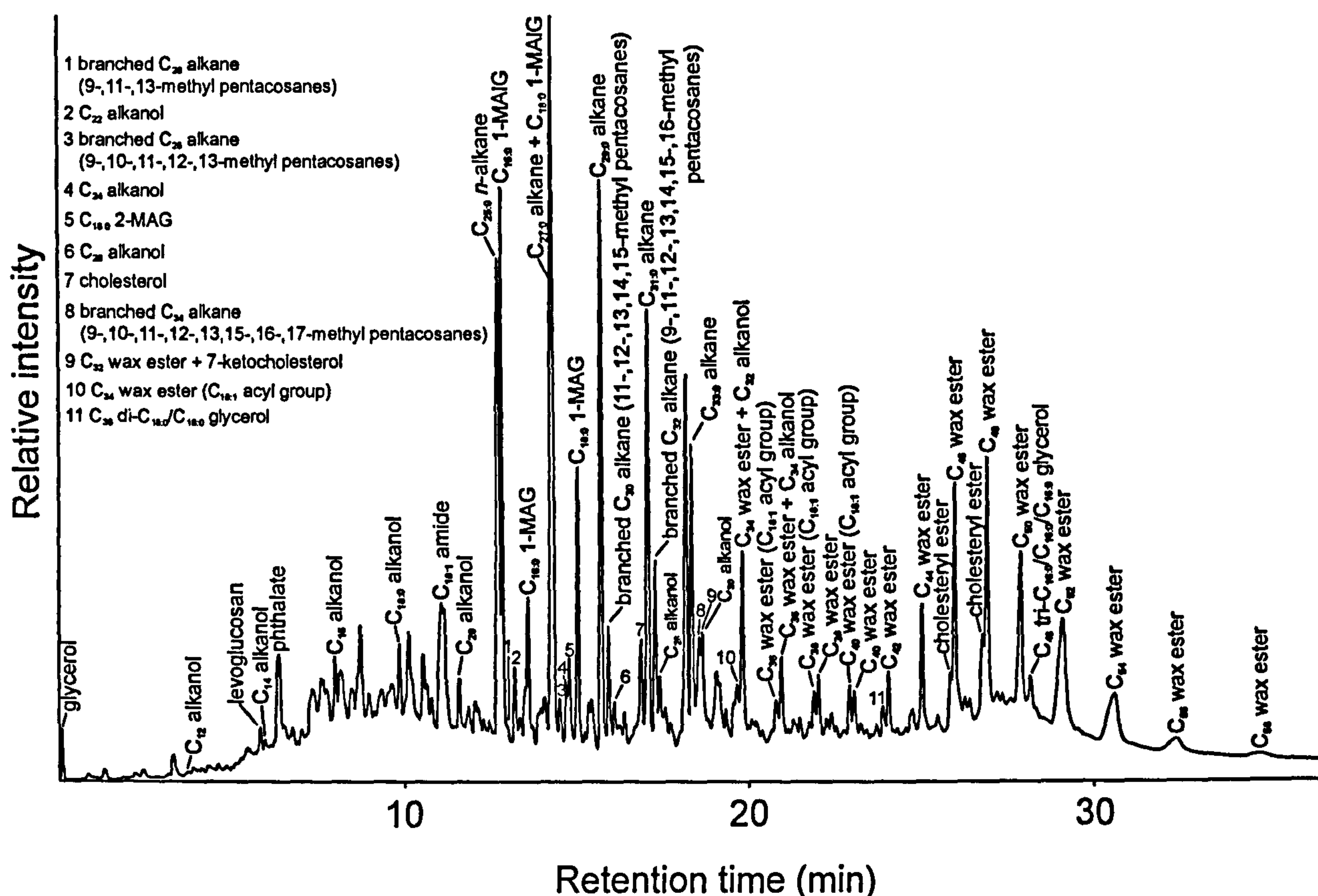


Figure 4.12a Total ion chromatogram from the GC/MS analysis of the neutral fraction of packing material/wrapping from the right calf [4] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

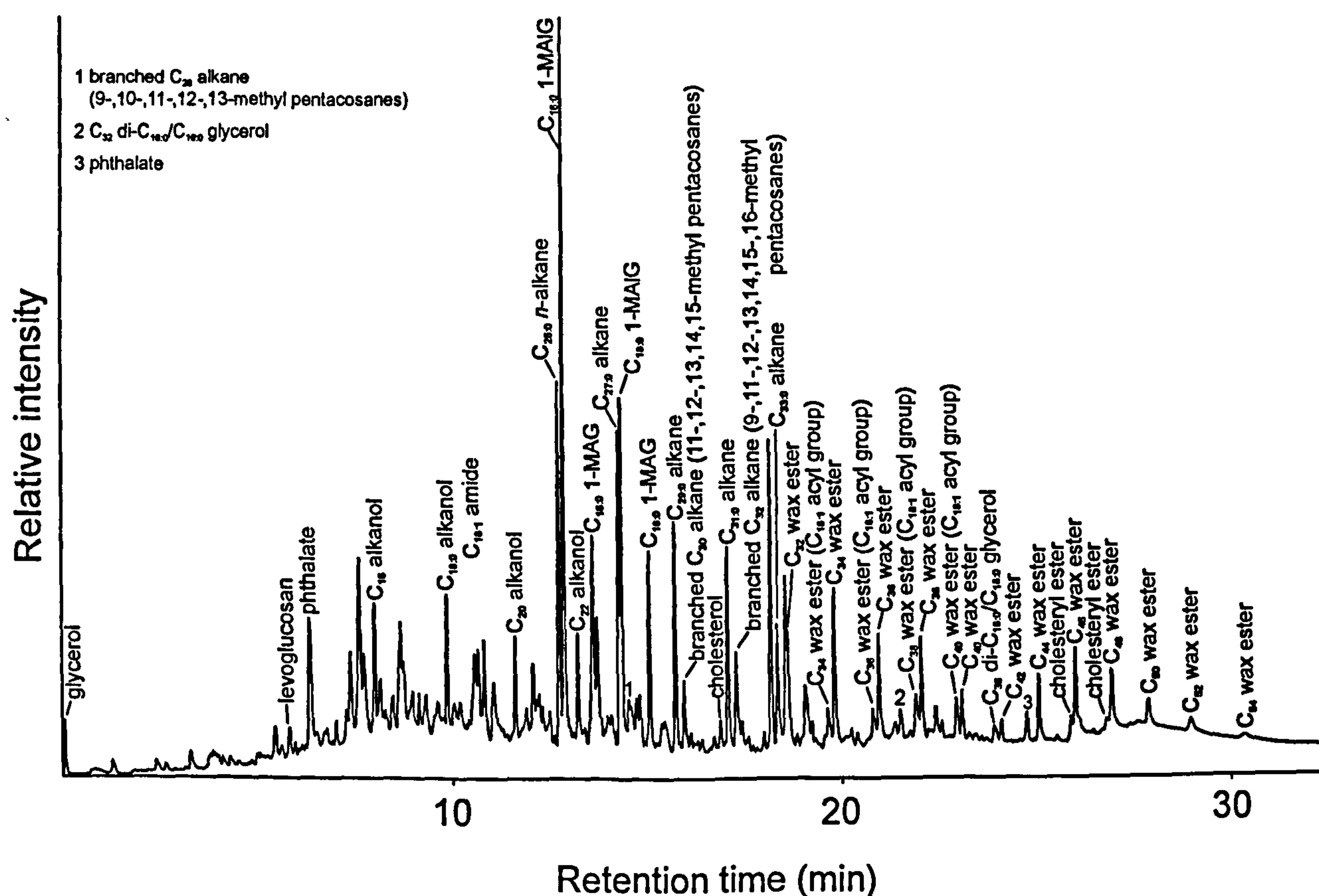


Figure 4.12b Total ion chromatogram from the GC/MS analysis of the neutral fraction of wrapping/tissue from the right calf [5] of the Theban priest ‘Horemkenesi’, XXIst dynasty (c. 1069-945 B.C.).

C_{18:0}/C_{18:0} diacylglycerol and the C₄₈ tri-C_{16:0}/C_{16:0}/C_{16:0}-glycerol. Cholesterol, 7-ketocholesterol (as their TMS ether), plus two cholesteryl esters were also identified as significant components, as were the C₁₂ to C₃₄ alkanols (even carbon numbers) (m/z 75, 103 and $[M-15]^+$) and the C_{18:1} amide (as the free compound, characterised by m/z 59, 72 and M^+ 281). The neutral components constituted ~20% w/w of the solvent extractable material.

4.4.4.5 Piece of 'resin'-soaked wrapping from left ankle/talus [1] (H7386/839)

The analysis of the neutral fraction by GC/MS, revealed no detectable neutral components, confirming that the solvent extractable portion of the sample consists essentially of free fatty acids.

4.4.4.6 Threads from wrappings from left ankle/talus [3] (H7386/839)

See 4.4.3.13 'total lipid extract'. The neutral components were relatively minor constituents of the extractable material which was dominated by carboxylic acids.

4.4.4.7 Wrapping fragments [2] (Ha7386/890)

The results for the neutral fraction analysed by GC/MS revealed very little solvent soluble material. The C_{16:0} monoglyceryl ether, C_{16:0} 1-monoacylglycerol and C₂₅ to C₃₃ *n*-alkanes with an odd-over-even predominance, were the only neutral components identified. These alkanes were also observed in the TD-GC/MS analysis. As stated, the neutral components were relatively minor constituents confirming that the solvent extractable portion of the sample consists essentially of free fatty acids.

4.4.4.8 Wrapping/tissue from right calf [5] (7386/760)

The results for the neutral fraction analysed by GC/MS are shown in Figure 4.12b. The chromatogram was dominated by a series of *n*-alkanes (C₂₅ to C₃₃) with an odd-over-even preference, together with appreciable amounts of wax esters in the C₃₂ to C₅₄ carbon number range, C₄₆ predominating. The C₃₂ to C₅₀ wax esters (even carbon numbers) contained predominantly the C_{16:0} acyl group, although others, particularly the C_{18:0}, were present. In contrast, the C₅₂ and C₅₄ wax esters (even carbon numbers) were found to contain predominantly the C_{20:0} acyl group, with significant amounts of the C_{22:0} acyl group. Also present were a series of C₃₄ to C₄₀ wax esters (even carbon numbers) with exclusively the C_{18:1} acyl moiety. A series of branched chain alkanes (C₂₈ to C₃₂), maximising at C₃₂, with an even-over-odd preference were also significant components.

These branched chain alkanes contain one methyl branch mid-chain, between the 9- and the 17- positions, with the odd numbered positions predominant. Of the glycerol derivatives identified, the C_{16:0} and C_{18:0} 1-*O*-monoalkylglyceryl ethers were major components of the neutral fraction, with moderate amounts of the C_{16:0} and C_{18:0} 1-monoacylglycerols, and lesser quantities of the C₃₂ di-C_{16:0}/C_{16:0} and C₃₆ di-C_{18:0}/C_{18:0} diacylglycerols. Cholesterol, plus two cholesteryl esters were also detected in moderate abundance, with more appreciable amounts of the C₁₆ to C₂₄ alkanols (even carbon numbers). The neutral components constituted <1% w/w of the solvent extractable material.

4.4.4.9 Muscle fibres from head of right femur [7] (Ha7386/945)

The results for the neutral fraction analysed by GC/MS revealed no neutral components, confirming that the solvent extractable portion of the sample consists essentially of free fatty acids.

4.4.4.10 'Resinous' material/muscle tissue from left hip/base of spine [8] (Ha7386/948)

The results for the neutral fraction analysed by GC/MS revealed a number of compound classes, including methyl esters, monoalkylglyceryl ethers, monoacylglycerols and small amounts of steroidal components. The major neutral compound was the bis-TMS ether C_{18:1} 1-monoacylglycerol, with the 1-monoacylglycerols containing C_{14:0}, C_{16:0} and C_{16:1} moieties present as major components. Observed in almost equal abundance to the major 1-monoacylglycerols were the C_{16:0} and C_{18:0} 1-*O*-monoalkylglyceryl ethers (as their bis-TMS ethers). Also dominant in the neutral fraction were the methyl esters of the carboxylic acids C_{16:0}, C_{18:0}, C_{18:1} and C_{18:0} 9,10-dihydroxy compound. Cholesterol and cholesta-3,5-dien-7-one were present in low abundance. The neutral fraction of this sample represents an almost insignificant portion of the total extractable lipid. However, the relatively high abundance of the 1-monoacylglycerols compared to the relatively small amounts of cholesterol and cholesta-3,5-dien-7-one agrees well with the results of steroidal compounds in the sample.

4.4.4.11 'Resinous' material from left side of upper spine [15] (Ha7386/908)

The results for the neutral fraction analysed by GC/MS are shown in Figure 4.13. Minor quantities of the C_{16:0} 1-monoacyl- and C_{16:0} 1-*O*-monoalkylglycerols (as their bis-TMS ethers) were identified, indicating almost complete hydrolysis of the original acyl lipids that would have dominated the tissues at the time of death (if indeed they do originate from

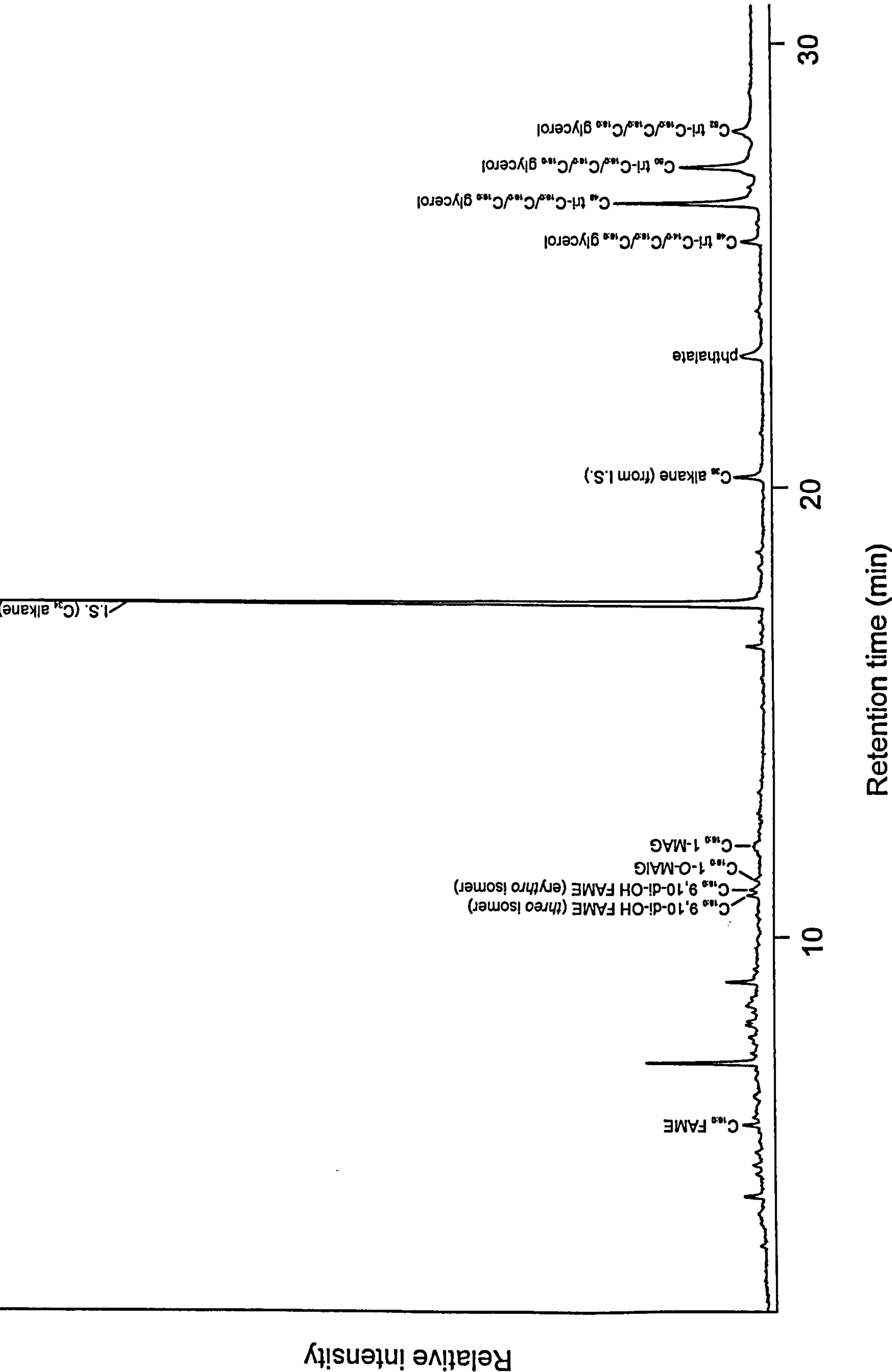


Figure 4.13 Total ion chromatogram from the GC/MS analysis of the neutral fraction of 'resinous' material from the left side of the upper spine [15] of the Theban priest 'Horemkenesi', XXIst dynasty (c. 1069-945 B.C.).

the body). Comparable amounts of the C_{16:0} and C_{18:0} 9,10-dihydroxy (*threo* and *erythro* isomers) fatty acid methyl esters were also observed. Eluting at later retention times (25.5 to 28.0 min), four triacylglycerols C₄₆ tri-C_{14:0}/C_{16:0}/C_{16:0}-glycerol, C₄₈ tri-C_{16:0}/C_{16:0}/C_{16:0}-glycerol and C₅₀ tri-C_{16:0}/C_{16:0}/C_{18:0}-glycerol and C₅₂ tri-C_{16:0}/C_{18:0}/C_{18:0}-glycerol were observed as the major components of the neutral fraction. The relative distribution of the acyl groups reflects the relative abundance of the free fatty acids identified in the acid fraction. The neutral fraction constitutes a significant proportion (~5% w/w) of the total lipid extracts of the sample.

4.4.4.12 'Resin' around the mouth [12] (Ha7386/686)

The analysis of the neutral fraction by GC/MS, revealed no detectable neutral components, confirming that the solvent extractable portion of the sample consists essentially of free fatty acids.

4.4.4.13 Whitish 'efflorescence' from head of right femur (a) [6] (Ha7386/945)

See 4.4.3.13 'total lipid extract'.

4.4.4.14 Whitish 'efflorescence' from head of right femur (b) [17] (Ha7386/945)

See 4.4.3.14 'total lipid extract'.

4.4.4.15 Whitish 'efflorescence' from head of right femur (c) [18] (Ha7386/945)

See 4.4.3.15 'total lipid extract'.

4.4.5 GC/MS – FAME DMDS derivatives – determination of double bond position in unsaturated fatty acids

4.4.5.1 Packing material/wrapping from right calf [4] (7386/760)

The results for the DMDS FAME derivatives analysed by GC/MS revealed the double bond positions in the monounsaturated fatty acids observed in the acid fraction (C_{16:1} and C_{18:1}). The C_{16:1} fatty acid consisted predominantly of the Δ^9 isomer (characterised by *m/z* 157, 173, 189 and M⁺ 362), with a small amount of the Δ^7 isomer (*m/z* 145, 185, 217 and M⁺ 362) and the Δ^{11} isomer (*m/z* 117, 213, 245 and M⁺ 362) present as a trace component. The major monounsaturated acid C_{18:1} consisted largely of the *cis* Δ^9 (oleic) isomer (*m/z* 173, 185, 217 and M⁺ 390) with ~10% w/w of the Δ^{11} isomer (*m/z* 145, 213,

245 and M^+ 390) and a small amount of the *trans* Δ^9 (elaidic) isomer (m/z 173, 185, 217 and M^+ 390).

4.4.5.2 'Resinous' material/muscle tissue from left hip/base of spine [8] (Ha7386/948)

The results for the DMDS FAME derivatives analysed by GC/MS revealed the double bond positions in the monounsaturated fatty acids observed in the acid fraction ($C_{16:1}$ and $C_{18:1}$). The $C_{16:1}$ fatty acid consisted predominantly of the Δ^9 isomer, with a small amount of the Δ^7 isomer, the major monounsaturated acid $C_{18:1}$ being present largely as the *cis* Δ^9 (oleic) isomer with ~10% w/w of the Δ^{11} isomer.

4.4.5.3 Packing material (natron?) between wrappings and skin of left(?) upper forearm [9] (Ha7386/731)

The results for the DMDS FAME derivatives analysed by GC/MS revealed the double bond positions in the monounsaturated fatty acids observed in the acid fraction ($C_{16:1}$ and $C_{18:1}$). The $C_{16:1}$ fatty acid consisted predominantly of the Δ^9 isomer, with a small amount of the Δ^7 isomer, the major monounsaturated acid $C_{18:1}$ being present largely as the *cis* Δ^9 (oleic) isomer with, again ~10% w/w of the Δ^{11} isomer.

4.4.5.4 'Resinous' material from left side of upper spine [15] (Ha7386/908)

The results for the DMDS FAME derivatives analysed by GC/MS revealed the double bond positions in the monounsaturated fatty acids observed in the acid fraction ($C_{16:1}$ and $C_{18:1}$). The $C_{16:1}$ fatty acid consisted predominantly of the Δ^9 isomer, with the major monounsaturated acid $C_{18:1}$ being present largely as the *cis* Δ^9 (oleic) isomer with a small amount of the Δ^{11} isomer.

It should be noted that mass chromatograms were obtained for all samples in order to ascertain whether diterpenoids (m/z 239, 251, 253), triterpenoids (m/z 163, 189, 203) and steranes (m/z 217) and hopanes (m/z 191) were present in trace amounts.

4.5 DISCUSSION

Within the material types the samples were relatively similar to each other, regardless of the part of the body from which they were taken. There were, however, marked differences between the packing, wrappings, etc. Yet all the samples analysed were found to contain

degraded acyl lipids, predominantly free fatty acids. None of the more exotic commodities connected with embalming were present in these samples, although the alkanes, with an odd-over-even preference, and wax esters found in a number of samples are indicative of a plant wax. Notably however, no di- or triterpenoids, characteristic of true or gum resins, or steranes and hopanes confirming a bitumen presence were identified, despite the fact that tree resins have been reported previously as having been utilised in the mummification of Horemkenesi (Taylor 1995, p.92). In fact the findings of the chemical analysis are in agreement with the results reported here, there having been no evidence for the di-or triterpenoids characteristic of tree resins and the previous analysis also identifying compounds indicative of degraded acyl lipids.

The packing material was expected to be largely inorganic, and the relatively small amount of extractable material present is consistent with this. Short chain fatty acids were abundant in these packing materials, as were γ - and δ -lactones and short chain hydroxy fatty acids, indicating that the lipids present in the fat or oil have undergone a high degree of oxidation. These samples do not appear to have undergone autoxidation, which usually predominates in oxidised lipids (Frankel 1999). The absence of the 11-hydroxyoctadecenoic acid in two of these packing materials [9] and [10] indicates a process with a degree of specificity. Notably, the 11-hydroxy acids is, with the 8-hydroxy isomer, one of the two major isomers produced by autoxidation. The predominance of the 9-hydroxy isomer (>80% w/w) in the packing/wrapping [4], with no 11-hydroxy isomer, strongly indicates enzymic oxidation which would result in the production of one specific isomer. The short chain fatty acids have not yet been fully elucidated, but may derive from the oxidation of polyunsaturated fatty acids which would have been present in the original fat/oil.

Notably, the lactones observed in the TD profile were not present in the acid fractions of the packing materials in any significant abundance. However, this is likely to be as a result of ease in which the lactone ring in these compounds opens and closes. On heating, these '4-hydroxy-' and '5-hydroxy-' carboxylic acids undergo ring closure, although this is a reversible process (Finar 1973, p.469-470). Ironically, the heat applied during the trimethylsilylation to aid derivatisation would be expected to cause this ring closure, hindering, in the case of the 5-hydroxy acids, and preventing, in the case of the 4-hydroxy acids, the conversion of these lactones to their TMS derivatives. On cooling the lactone ring can open making its subsequent analysis difficult. A notable study reported finding

“every positional isomer except the 4-hydroxy acids” in a naturally mummified body from Egyptian Nubia (Gulaçar et al. 1989, p.61-72). Heating these compounds in the probe during TD at 310°C clearly results in the expected lactonisation of these compounds, and presumably they are sufficiently stable to be identified in this form in the TD-GC/MS analysis. These packing materials also contained a large amount of polymeric material, consisting of ketones indicative of polymeric acyl lipids, with the C₁₇ homologue dominating in the mud packing [11] and packing/wrapping [4], and deriving from the C_{16:0} fatty acid, a major component of (particularly degraded) lipids. Also abundant however, were the mid-chain ketones C₁₀ to C₁₈. Mid-chain ketones have been observed in pottery vessels, and have been shown to result from the heating (and subsequent dehydration and decarboxylation) of fatty acids, the major compounds being the C₃₁ to C₃₅ alkanones deriving from C_{16:0}/C_{16:0}, C_{16:0}/C_{18:0} and C_{18:0}/C_{18:0} fatty acids dominant in the acyl lipids (Raven et al. 1997, p.267-285). The results obtained here are consistent with this, the major constituents 7-pentadecanone and 8-pentadecanone deriving from the C_{7:0}/C_{9:0} and C_{8:0}/C_{8:0} fatty acids respectively. The relative distribution of these mid-chain ketones mirrors the relative abundances of the short chain fatty acids present in these samples (see Fig. 4.14). Their production in the probe in any major quantities can be excluded given the low abundance of the free fatty acids in TD, particularly in the mud packing [11], with no free acids in the TD yet abundant mid-chain ketones in the Py-GC/MS analysis. The packing material has not been analysed to date, but is believed to be natron. Given that thermal processes were almost certainly part of the embalming process, and that the natron could serve to catalyse the reaction by both acting as a reaction site for the fatty acids and as a dehydrating agent, the presence of these ketones could be expected.

The wrapping samples consisted of palmitic acid with significant amounts of the monohydroxy-, dihydroxy- and diacids, indicative of the oxidation of plant oils (Serpico & White 1996, p.128-139). The high palmitic to stearic ratios (8.4, 3.3, 7.1 and 6.4) would indicate a plant oil. The linen threads [3] also contained a wax of some sort (possibly degraded beeswax), although its exact origin could not be determined. The resinous materials analysed were also similar, characterised by lower palmitic to stearic ratios (2.2 to 3.3) and a prevalence of dicarboxylic acids and the 9,10-dihydroxyoctadecanoic acid, with lesser amounts of the 8,10-, 9,11- and 11,12-dihydroxyoctadecanoic acids (the latter indicative of the C_{18:1} Δ^{11} isomer). The presence of this latter compound was confirmed by the analysis of the DMDS FAME derivatives. The whitish ‘effluorescence’ samples consisted largely of the C_{16:0} and C_{18:1} fatty acids, all three being very similar in

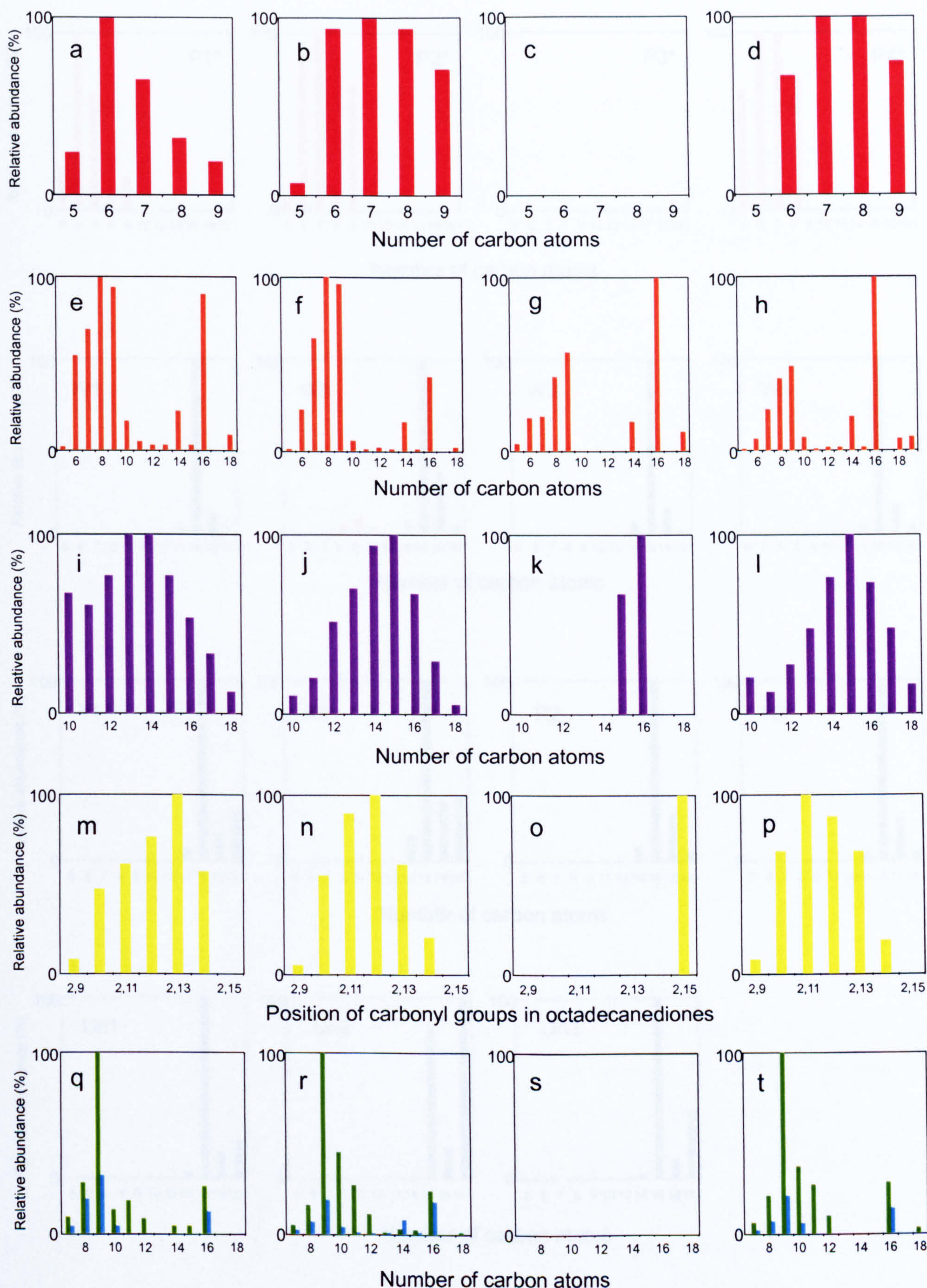


Figure 4.14 Histograms showing the distributions of: (i) fatty acids in the TD-GC/MS analyses of the packing materials ([9],[10],[11],[4]) from Horemkenesi (a to d); (ii) 2-alkanones in the Py-GC/MS analyses of the packing materials ([9],[10],[11],[4]) from Horemkenesi (e to h); (iii) mid-chain ketones in the Py-GC/MS analyses of the packing materials ([9],[10],[11],[4]) from Horemkenesi (i to l); (iv) 2,x-octadecanediones (x=9 to 15) in the Py-GC/MS analyses of the packing materials ([9],[10],[11],[4]) from Horemkenesi (m to p); (v) lactones [γ - (in green) and δ - (in cyan)] in the TD-GC/MS analyses of the packing materials ([9],[10],[11],[4]) from Horemkenesi (q to t). [see Table 4.2]

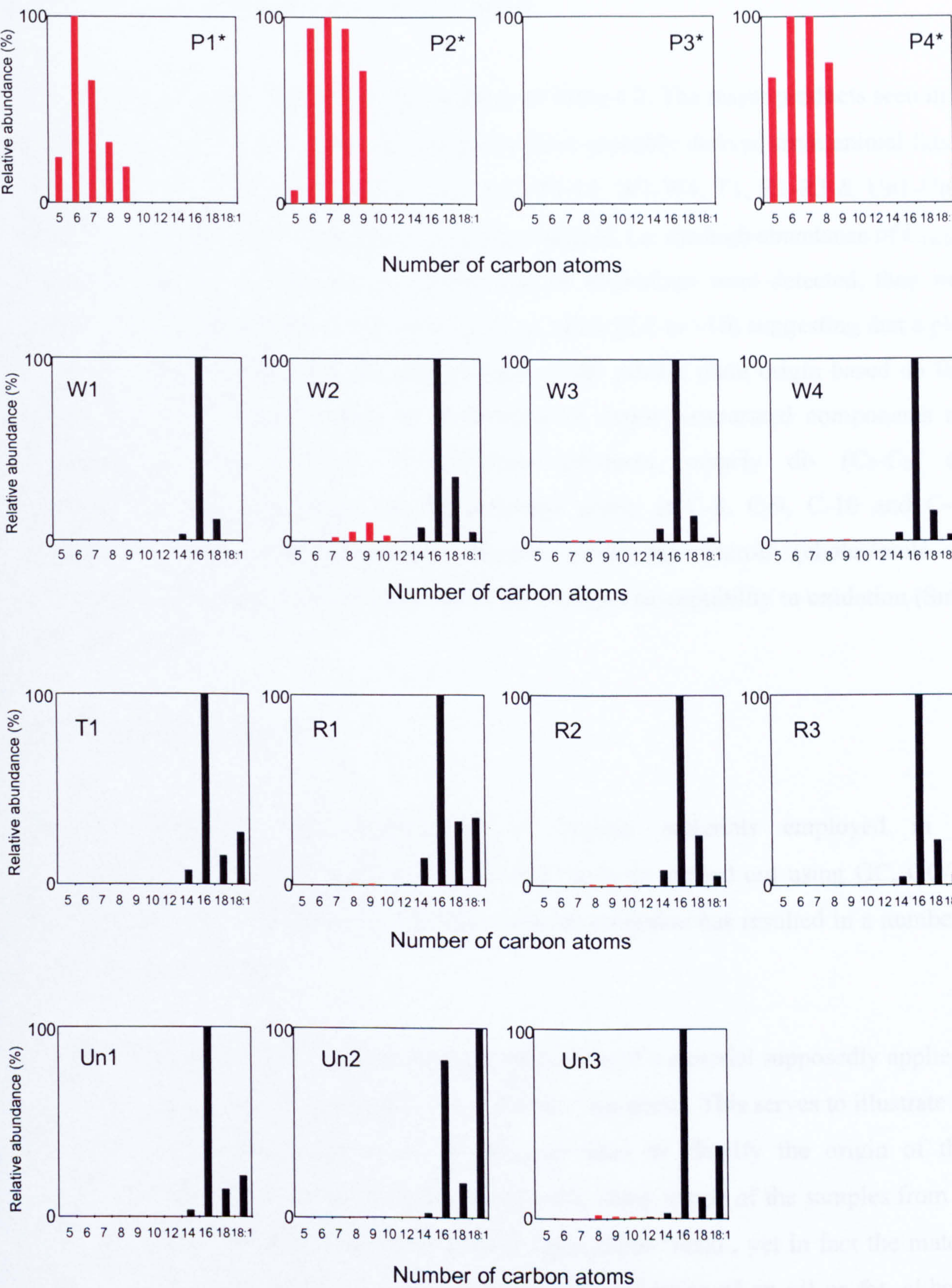


Figure 4.15 Histograms showing the distributions of fatty acids in samples of packing [P1-P4], wrapping [W1-W4], tissue [T1], ‘resin’ [R1-R3] and unknowns [Un1-Un3] from the Theban priest Horemkenesi [see Table 4.2]. Volatile fatty acids observed in the TD-GC/MS analyses are in red, fatty acids observed in the GC/MS analyses [total lipid extracts and acid fractions] in black. TD values are corrected to give the equivalent solvent extract abundances. * Very little solvent extractable material present.

composition. The alkanes and wax esters identified in the packing/wrapping [4] confirm a wax origin as a significant portion of this sample.

A summary of the results of this study is given in Table 4.2. The major products seen in all the samples analysed are degraded acyl lipids, most probably derived from animal fats or plant oils. In all of the 15 examples, (Fig. 4.15 P1-P4, W1-W4, T1, R1-R3 & Un1-Un3), the fatty acid distributions strongly suggest plant origins, i.e. the high abundance of C_{16:0} as compared with C_{18:0}. Although cholesterol and its derivatives were detected, they were present only in trace amounts, with the C_{16:0}/C_{18:0} ratios (2.2 to >10) suggesting that a plant origin is likely. Beyond this, the identification of the precise plant origin based on fatty acids alone is not possible, due to degradation of major unsaturated components and evidenced by the presence of oxidations products, namely di- (C₇-C₉) and hydroxycarboxylic acids (C₁₈ with the hydroxyl group at C-8, C-9, C-10 and C-11, indicative of both autoxidation and enzymic oxidation). Sterols (cholesterols and phytosterols) are absent in most cases due to their known susceptibility to oxidation (Smith 1996, p.453-469).

4.6 CONCLUSIONS

The characterisation and identification of organic materials employed in the mummification of the XXIst priest Horemkenesi has been carried out using GC, GC/MS and sequential TD-GC/MS and Py-GC/MS. This investigation has resulted in a number of significant conclusions.

- (i) Confirmation of the erroneous identification of a material supposedly applied to the body of Horemkenesi as a so-called 'tree resin'. This serves to illustrate that visual appearance can in no way be used to identify the origin of these amorphous organic materials. The brown, shiny nature of the samples from this mummy fits the all-too-often-used descriptions 'resin', yet in fact the material consisted largely of degraded acyl lipids indicative of an oil or fat, although plant waxes may have been utilised.
- (ii) Sequential TD-GC/MS and Py-GC/MS have shown their value in aiding the identification of a complex suite of lipids, TD providing a useful 'fingerprint', comparable with the more conventional solvent extracts. However,

polyfunctional components were not observed, and given the likely abundance of such polar molecules in aged samples this is a notable limitation.

- (iii) Bitumen was not present in these samples, as demonstrated by the absence of any bitumen markers in any of the samples analysed, despite searches using mass chromatograms for the steranes and hopanes characteristic of natural bitumens.
- (iv) Resins were not present in these samples, as demonstrated by the absence of any terpenoids markers in any of the samples analysed, despite searches using mass chromatograms for the diterpenoids and triterpenoids characteristic of these commodities.
- (v) A number of pathways are involved in the oxidation of these lipids, including autoxidation and enzymic oxidation, the later presumed to be facilitated by the presence of bacteria in these samples.
- (vi) The chemical composition of the organic component in each of the sample types (i.e. wrapping, packing, 'resins', tissues), was notably similar regardless of their original location on the body.

CHAPTER 5

Human mummies: an historical comparison

CHAPTER 5: HUMAN MUMMIES - AN HISTORICAL COMPARISON

5.1 OBJECTIVES

This chapter involves the chemical investigation of a number of human mummies in an attempt to characterise and identify the organic materials employed in their embalming. Significant numbers of samples (tissues, wrappings, 'resin/bitumen') were taken from provenanced and dated mummies, with the investigation largely focussing on examples from the 'classic' mummy-making era of pharaonic/dynastic Egypt in an attempt to understand something of the chronological development of mummification. GC, GC/MS and sequential TD-GC/MS and Py-GC/MS were utilised to facilitate the characterisation and identification of the organic embalming agents employed over a three millennia period.

5.2 INTRODUCTION

There have been surprisingly few studies carried out to determine the organic embalming agents employed in ancient Egyptian mummification (see 2.4), even fewer of them having used the methods of chemical analysis (GC/MS, etc.) capable of identifying such complex degraded residues. Yet none of even these few studies have looked at more than one provenanced and dated pharaonic mummy, with the exception of a single study carried out at Bristol (Buckley et al. 1999, p.443-452). The study presented here examines samples from a total of 14 provenanced and dated mummies, 8 of which date to the pharaonic/dynastic period and which were selected as examples of 'classic' Egyptian embalming rather than the later Graeco-Roman efforts which inevitably differ from earlier attempts and serve to obscure the ancient origins of mummification. Factors such as sex, status and location on the body were also taken into account wherever possible.

The human mummies included in this study are shown in Table 5.1, together with a description of the samples analysed. A multiple sampling approach, the importance of which was demonstrated by Serpico and White (Serpico & White 1998, p.1037-1048), was undertaken wherever possible, using at least two samples from each mummy, with scope for further future analysis given the necessary time constraints involved in the current study. Samples were also taken from as many different locations on the body as possible,

Table 5.1. Provenance and date of human mummies studied and the origin of samples analysed.

Mummy	Date/age	Provenance	Sample location and description ^a
Male adult ^a f H640 1	2686-2613BC (III rd dynasty)	Medum	'Resin'-soaked wrapping from hip/innominate bone [1] 'Resin/tissue' from hip/innominate bone [2] 'Resin/tissue' (loose) on thigh/femur [3]
Male adult ^b (<i>'Khnumnakht'</i>) f 21471 2	1985-1795BC (XII th dynasty)	Rifeh	'Resin/tissue'/ bandaging [1]
Female adult ^c f 1909.527 3	1650-1550BC (XVII th dynasty)	Qurna, Thebes	'Resin/tissue' from head of right tibia [1] Textile material/'fatty matter' from mass of cloth [2]
Child (sex ?) ^c f 1909.527 4	1650-1550BC (XVII th dynasty)	Qurna, Thebes	Unspecified bone and cartilage [2] Stained wrapping [3]
Head (sex ?) ^d f 1976.159.267 5	1550-1069BC (XVIII th – XX th dynasties)	Thebes	'Resin/skin' beneath right eye/orbit [1] 'Skin/resin' from back of cranium [3]
Female(?) adult ^e f EA74303 6	1069-664BC (XXI st -XXV th dynasties)	Thebes?	'Resin' from thoracic cavity [1]
Female adult ^a (<i>'Neskhons'</i>) f H 5062 7	945-715 BC (XXII nd dynasty)	Thebes	'Resin'-soaked outer wrapping from left side of neck/ cervical vertebrae [1] 'Resin'-soaked wrapping from left hip/innominate bone [2]
Male adult ^d (<i>'Pedeamun'</i>) f 1953.72 8	664-404 BC (XXVI th -XXVII th dynasty)	Thebes	'Resin' from inside of cartonnage at back of cranium [1] 'Resin' from top of cranium [3] 'Resin' from upper edge of cartonnage, opposite right cheek/zygomatic bone [5]
Female adult ^c f 1956.352 9	332-30 BC (Ptolemaic)	Thebes	'Resin' attached to linen thread on right ankle/talus [1] 'Resin' from top of cranium [2]
Male adult (1) ^c f 1911.2101 10	30BC -395AD (Roman)	Hawara	'Resin'-soaked outer wrapping below right scapula [1] 'Resin'-soaked wrapping from front at base of right side of thorax [3]
Male adult (2) ^c f 1911.2102 11	30BC - 395AD (Roman)	Hawara	'Resin' from side/base of left foot [1] 'Resin' from left strip of wrapping at base of golden mask [3]
Male child (wrapped) ^c f 1956.357b 12	30BC - 395AD (Roman)	Thebes	'Resin' on outer wrapping from area between calves [1] 'Resin'-stained wrapping from back of mummy at base of thorax [2]
Male child (unwrapped) ^c f 1956.357c 13	30BC - 395AD (Roman)	Thebes	'Resin' from abdominal cavity (kidney area)

^a Bristol Museum; ^b Manchester Museum; ^c National Museum of Scotland; ^d Liverpool Museum; ^e British Museum; ^f museum number; ^g the term 'resin' denotes physical appearance and does not presuppose any chemical composition or biological origin.

including human tissue, wrappings and 'resin'-like material, in order to obtain as full a picture as possible and to determine whether a number of different commodities were applied to the different body parts as suggested by literary texts, e.g. the Ritual of Embalming (see Chapter 1). This seems to have been the case with the head and area around the embalming incision, where symbolic unguents may have been applied. Obviously the greater number of samples analysed the more accurate and meaningful the picture obtained, although aforementioned time constraints meant that only two samples could be analysed from most of the mummies, with each sample chosen to give the most meaningful information within these limits.

Given the sensitive nature of the material concerned, the sampling of human remains must necessarily be pragmatic and therefore concentrated on areas of the mummies already accessible/exposed and/or damaged. These generally tend to be the back of the head, shoulder blades and base of the spine, all areas where decomposition is greater as a result of pressure caused by the body laying on its back in contact with the coffin (see 2.1). Yet if the remains are pre-New Kingdom (pre-1550 BC) in date, when the body was generally laid on its left side, the most damaged areas will correspondingly be along the left side of the body. If the date of the body can therefore be ascertained prior to sampling, the areas of potential damage may be easily located and convenient samples taken. Otherwise, general damage to the bodily extremities such as the feet are alternative sources of samples. Yet care was taken to avoid potentially contaminated areas already exposed, and sample sizes taken reflected the need to obviate any problems of this nature, in addition to difficulties associated with highly oxidised/degraded material on the surface of the resins, waxes, fats, etc.

Diagnostic marker compounds present in the original embalming agents and resistant to degradation can be related to specific embalming agents and were therefore used to identify the 'balms'. GC/MS and TD/Py-GC/MS were used to facilitate the molecular separation and identification of these marker compounds. Due to the nature and history of the proposed embalming materials both free and polymerised components were likely to be present (see 2.5). Therefore the 'dual' approach of GC/MS (following solvent-extraction procedures) and sequential TD-GC/MS and Py-GC/MS was employed to allow the characterisation and identification of both the free (solvent-extractable) marker compounds and the recognisable sub-units of polymeric materials not amenable to the more conventional GC/MS approach.

5.3 BACKGROUND

The potential origin of the organic embalming agents has been discussed above (Chapter 1), together with the archaeological and historical background of these materials. In summary, true resins (conifer and Pistacia), gum (e.g. frankincense and myrrh) and balsamic (e.g. storax) resins, beeswax, bitumen, animal fats, plant oils and others (Tables 1.3, 2.1) are all possible sources of embalming materials. Yet the indiscriminate use of the terms 'resin' and 'bitumen' perpetuate the extreme ignorance surrounding the nature of the organic materials used in mummification, a situation the current study seeks to begin to rectify.

The diverse chemistry of the organic embalming materials has also been described above (Chapter 2, 2.3) and so will not be detailed here. The chemistry of each of the samples analysed will be dealt with in turn in the results section and related back to their likely origin. In general, degradation processes such as dehydration, aromatisation, polymerisation and particularly oxidation are likely to have taken place over the 1,500 to 4,500 years the samples remained *in situ*. Notably, only two of the four provenanced and dated studies carried out outside Bristol mention the identification of highly oxidised components in the organic materials they analysed, the first identifying oxidised diterpenoids (Koller et al. 1998, p.343-344) and the second mentioning oxidised triterpenoids, although their exact nature is not discussed (Colombini et al. 2000, p.19-29). Given the embalming agents' likely treatment (e.g. heating, etc.) and their likely post-depositional environment, this is something of a serious omission, since it is likely that oxidised constituents will be present in appreciable abundance (Serpico & White 1998, p.1037-1048; Proefke et al. 1992, p.105.A-111.A; Weser et al. 1998, p.511.A-516.A; Buckley et al. 1999, p.443-452).

Interpretations must be made with circumspection, given that mixtures may well have been used (Herodotus II, 83-90, 1954, p.160-161; Diodorus in Smith & Dawson 1924, p.62-63), and an open mind is a crucial prerequisite if the picture obtained is to be at all meaningful.

5.4 RESULTS

The focus of this study was the characterisation and identification of the organic embalming agents employed in each of the mummies investigated. A further important aspect of the research was to assess the value of sequential TD/GC-MS and Py-GC/MS which is of particular value given the small sample sizes required (~0.1 mg) and the limited sample preparation involved. A comparison of the data obtained from each technique for a given sample and mummy will be presented in order to allow a direct comparison of the data obtained by each technique, particularly a comparison between TD-GC/MS and the more conventional GC/MS. The subsequent discussion will then where appropriate make general comparisons of the findings and the relative merits of the techniques utilised, together with the implications of the information they have provided.

For a summary of the findings of this study see Table 5.2. The identification of the compounds observed was based on both their mass spectra (NIST/EPA/NIH Mass Spectral Database) and retention times. For TD/Py-GC/MS the compounds are present as the free compounds. The compounds identified in the solvent extracts (acid and neutral fractions) are present as the free compounds or as their TMS derivatives.

5.4.1 Male Adult, IIIrd dynasty (c.2686-2613 BC), Old Kingdom, Medum (H.640)

5.4.1.1 'Resin'-soaked wrapping from hip/innominate bone [1]

TD-GC/MS

TD-GC/MS analysis revealed a homologous series of *n*-alkanes (characterised by ions of *m/z* 57 and 71) from C₂₀ to C₃₁, maximising at C₂₇ (M⁺ 380) with small amounts of branched alkanes (C₂₄ to C₃₁, maximising at C₂₉ (M⁺ 408)). There was no odd-over-even preference, as observed in plant derived waxes, with even carbon numbers equally abundant. No other components were present.

Py-GC/MS

Py-GC/MS analysis yielded a homologous series of *n*-alkanes from C₂₅ to C₃₃, with C₃₀ to C₃₂ dominant and maximising at C₃₁ (M⁺ 436). Lesser amounts of branched alkanes were also present (C₃₀ to C₃₃, maximising at C₃₂ (M⁺ 450)). There was no odd-over-even preference with even carbon numbers equally abundant. These hydrocarbons were the only components detected.

Table 5.2. Provenance and date of mummies, origin of ‘balms’ and their composition.

Mummy	Date/age	Provenance	Sample location and description ⁷	Inferred components of embalming “resin”	Relative abundance (%) ⁹
Male adult ¹ 6 H640 1	2686-2613BC (III rd dynasty)	Medum	‘Resin’-soaked wrapping from hip/innominate bone [1]	Paraffin wax Fat/oil	100 trace
			‘Resin/tissue’ from hip/innominate bone [2]	Paraffin wax Fat/oil	98.5 1.5
			‘Resin/tissue’ (loose) on thigh/femur [3]	Paraffin wax Fat/oil	98 2
Male adult ² (‘Khnumnakht’) 6 21471 2	1985-1795BC (XII th dynasty)	Rifeh	‘Resin/tissue’/ bandaging [1]	Fat/oil ^e Proteinaceous material	90 10
Female adult ³ 6 1909.527 3	1650-1550BC (XVII th dynasty)	Qurna, Thebes	‘Resin/tissue’ from head of right tibia [1]	Fat/oil ^e Coniferous resin Wax Balsam/umbelliferae ⁸ Proteinaceous material	89 0.3 1 0.4 9
			Textile material/‘fatty matter’ from mass of cloth [2]	Fat/oil Coniferous resin Wax	99 0.2 0.6
			Unspecified bone and cartilage [2]	Fat/oil ^b Coniferous resin? Wax Balsam/umbelliferae Proteinaceous material	71 trace? 2 16 10
Child (sex ?) ³ 6 1909.527 4	1650-1550BC (XVII th dynasty)	Qurna, Thebes	Stained wrapping [3]	Fat/oil Coniferous resin Wax Balsam/umbelliferae	97 0.3 1 1
			‘Resin/skin’ beneath right eye/orbit [1]	Fat/oil Coniferous resin Balsam/umbelliferae Proteinaceous material	97 0.4 1 2
Head (sex ?) ⁴ 6 1976.159.267 5 (contd)	1550-1069BC (XVIII th – XX th dynasties)	Thebes			

[illegible]

Female adult 9 (contd)	332-30 BC (Ptolemaic)	Thebes	'Resin' from top of cranium [2]	Fat/oil Pistacia resin Balsam/umbelliferae Beeswax Chinese insect wax	3 3 1 89 3
Male adult (1) ³ 6 1911.2101 10	30BC - 395AD (Roman)	Hawara	'Resin'-soaked outer wrapping below right scapula [1] 'Resin'-soaked wrapping from front at base of right side of thorax [3]	Fat/oil ¹ Coniferous resin Balsam (storax)/cassia? Beeswax ^{p,u} Fat/oil Coniferous resin Beeswax	78 16 trace 6 77 22 1
Male adult (2) ³ 6 1911.2102 11	30BC - 395AD (Roman)	Hawara	'Resin' from side/base of left foot [1] 'Resin' from left strip of wrapping at base of golden mask [3]	Fat/oil ^a Coniferous resin Balsam/umbelliferae Beeswax ^{q,v} Fat/oil Coniferous resin Balsam/umbelliferae Beeswax	35 37 0.2 27 25 44 0.2 30
Male child (wrapped) ³ 6 1956.357b 12	30BC - 395AD (Roman)	Thebes	'Resin' on outer wrapping from area between calves [1] 'Resin'-stained wrapping from back of mummy at base of thorax [2]	Fat/oil ^a Coniferous resin A sugar/gum? Fat/oil Coniferous resin	87 8 4 77 23
Male child (unwrapped) ³ 6 1956.357c 13	30BC - 395AD (Roman)	Thebes	'Resin' from abdominal cavity (kidney area)	Fat/oil ^p Coniferous resin Balsam/umbelliferae	87 13 0.1

¹ Bristol Museum; ² Manchester Museum; ³ National Museum of Scotland; ⁴ Liverpool Museum; ⁵ British Museum; ⁶ museum number; ⁷ the term 'resin' denotes physical appearance and does not presuppose any chemical composition or biological origin; ⁸ of the 16 cases where components indicative of a degraded balsam were found, 15 could not originate from the 'local' balsamic resin storax (*Liquidambar orientalis*); ⁹ % relative abundance based on absolute concentrations, calculated based on internal standards added at the extraction stage (TD taken into account where appropriate). ¹⁰ % relative abundance based on peak areas in TD profile. Superscript letters (a-v) refer to the histograms shown in Fig. 5.22.

GC/MS – Acid fraction

The results for this fraction, analysed by GC/MS, revealed that very little acidic material was present. Trace amounts of palmitic (C_{16:0} FA, as TMS ester [M-15]⁺313) and stearic (C_{18:0} FA, as TMS ester [M-15]⁺341) acids were the only components detected.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS displayed a homologous series of *n*-alkanes ranging from C₂₀ to C₄₀, maximising at C₂₅ (M⁺ 352), with small amounts of branched alkanes (C₂₄ to C₃₄, maximising at C₂₉). There was no odd-over-even preference, as observed in plant derived waxes, with even carbon numbers equally abundant. Although the abundances of the longer chain alkanes fell away less sharply, and the major *n*-alkane differs from that obtained for TD the overall distributions obtained are very similar.

5.4.1.2 'Resin/tissue' from hip/innominate bone [2].

TD-GC/MS

The results of the TD-GC/MS are shown in Figure 5.1a. It displays a homologous series of *n*-alkanes ranging from C₂₀ to C₃₁, maximising at C₂₅, with a small abundance of branched alkanes (C₂₄ to C₃₀, maximising at C₂₆ (M⁺ 366). There was no odd-over-even preference, as observed in plant derived waxes, with even numbers equally abundant. No other components were present.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.1a, inset. It displays a homologous series of *n*-alkanes from C₂₁ to C₃₃, with C₃₀ and C₃₂ dominant and maximising at C₃₁. Minor amounts of branched alkanes were also present (C₂₄ to C₃₃, maximising at C₃₂) at low abundance. There was no odd-over-even preference with even numbers equally abundant.

GC/MS – Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.1b, inset. Very little acidic material was in this fraction. Minor amounts of palmitic (C_{16:0} FA) and stearic (C_{18:0} FA) and barely detectable amounts of myristic (C_{14:0} FA, as TMS ester [M-15]⁺285) and oleic (C_{18:1} FA, as TMS ester [M-15]⁺339) acids were the only acidic components present.

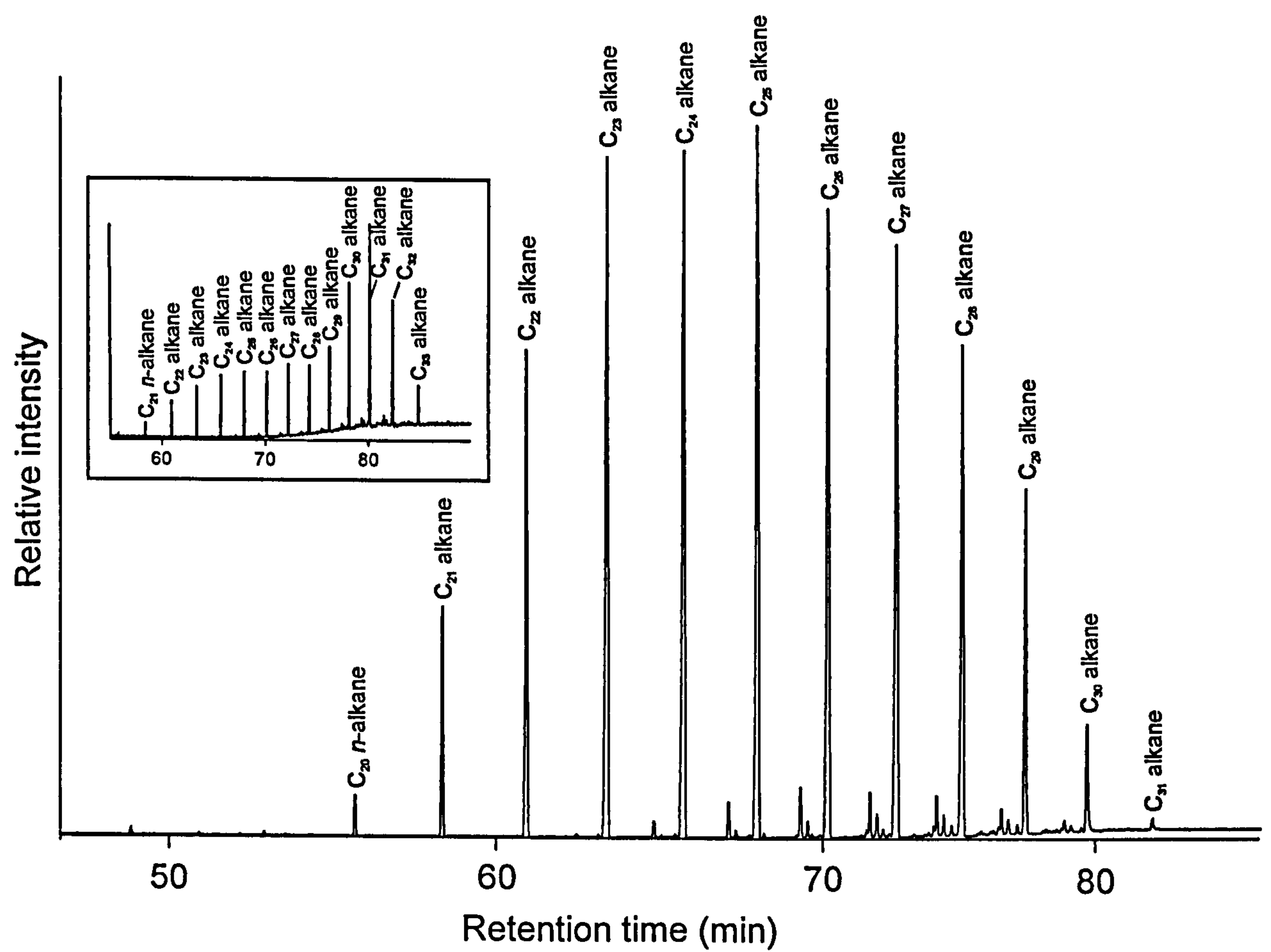


Figure 5.1a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of ‘resin/tissue’ from the hip/innominate bone [2] of the Old Kingdom male adult, IIIrd dynasty (c. 2686-2613 B.C.).

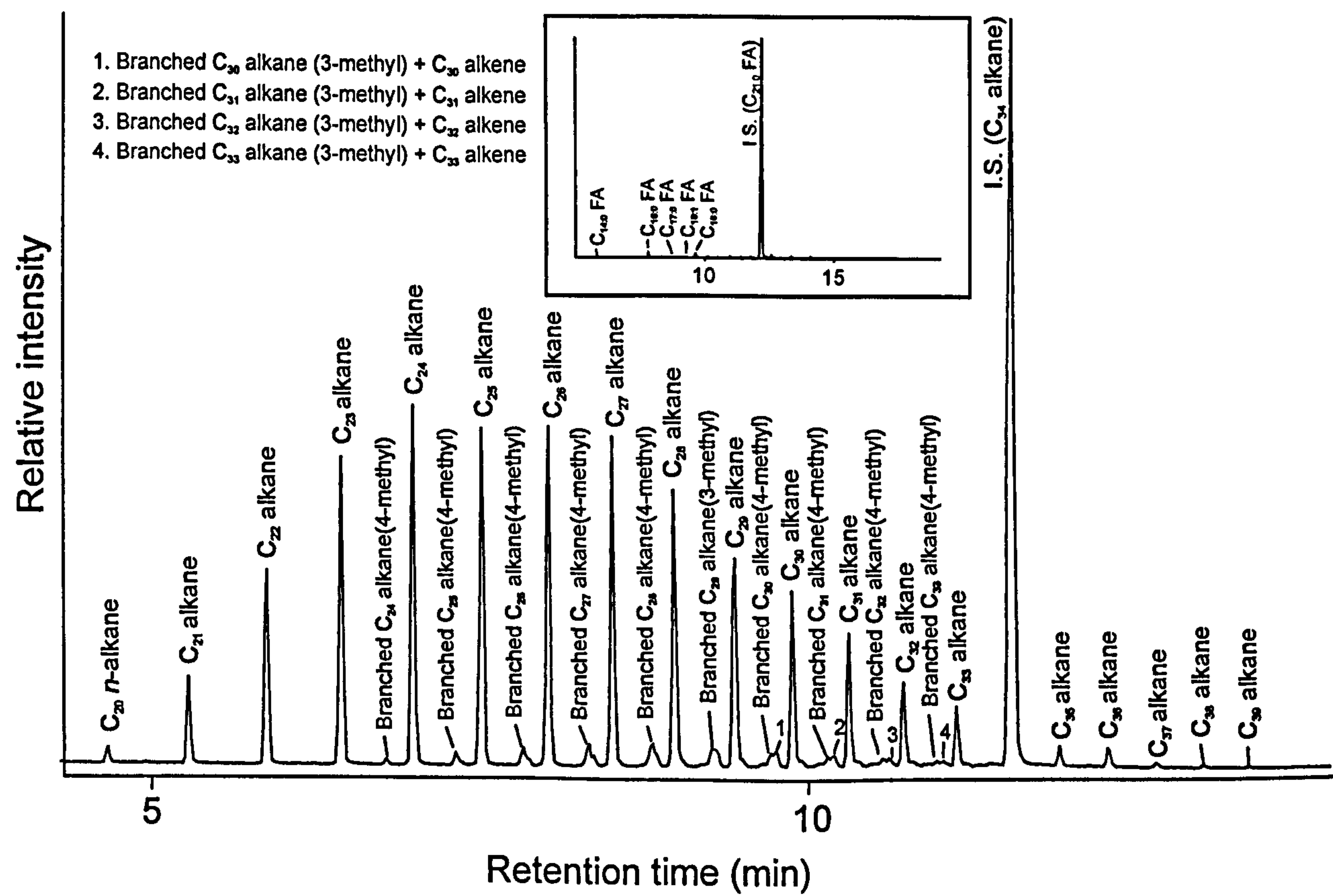


Figure 5.1b Total ion chromatograms from the GC/MS analyses of the neutral fraction, and (inset) acid fraction of ‘resin/tissue’ from the hip/innominate bone [2] of the Old Kingdom male adult, IIIrd dynasty (c. 2686-2613 B.C.).

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.1b. It displays a homologous series of *n*-alkanes ranging from C₂₀ to C₃₉, maximising at C₂₄ (M⁺ 338), with small amounts of branched alkanes (C₂₄ to C₃₃, maximising at C₂₈ (M⁺ 394). There was no odd-over-even preference with even numbers equally abundant. Although the abundances of the longer chain alkanes fall away less sharply, and the major *n*- and branched alkanes differ from that obtained for TD, the overall results are very similar.

5.4.1.3 'Resin/tissue' (loose) on thigh/femur [3].

TD-GC/MS

The results of the TD-GC/MS analysis revealed a homologous series of *n*-alkanes ranging from C₂₀ to C₃₀, maximising at C₂₆, with branched alkanes (C₂₄ to C₃₀, maximising at C₂₇) present in low abundance. There was no odd-over-even preference, again with even numbers equally abundant. No other components were present.

Py-GC/MS

The results of the Py-GC/MS analysis revealed a homologous series of *n*-alkanes ranging from C₂₃ to C₃₂, with C₂₉ to C₃₁ dominant and maximising at C₃₀ (M⁺ 422). Small amounts of branched alkanes were also present (C₂₉ to C₃₂, maximising at C₃₁). There was no odd-over-even preference with even numbers equally abundant.

GC/MS – Acid fraction

The results for the acid fraction analysed by GC/MS revealed that very little acidic material was present. Trace amounts of palmitic (C_{16:0} FA) and stearic (C_{18:0} FA) acids were the only acidic components detected.

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS revealed a homologous series of *n*-alkanes ranging from C₂₀ to C₄₀, maximising at C₂₆, with low abundances of branched alkanes (C₂₄ to C₃₄, maximising at C₂₉). There was no odd-over-even preference with even numbers equally abundant. Although the abundances of the longer chain alkanes fall away less sharply, and the major branched alkane differed from that obtained for TD the distributions are very similar.

5.4.2 Male Adult 'Khnumnakht', XIIth dynasty (c.1900 BC), Middle Kingdom, Rifeh (MM.21471)

5.4.2.1 'Resin/tissue'/bandaging

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.2a. The compounds detected included a series of fatty acids (C_7 to C_{18}), with the major components being $C_{16:0}$, $C_{18:1}$, $C_{18:0}$ and $C_{14:0}$ in order of decreasing abundance. Two steroidal components were observed as major components, eluting at later retention times (72.4 and 72.9 min). The earliest eluting of these was characterised by the presence of fragment ions m/z 141, 143, 247, 253 and M^+ 366 identifying it as cholesta-3,5,7-triene (see Figure 5.3.a). The later eluting peak was characterised by the presence of fragment ions m/z 145, 147, 247, 255, 260 and M^+ 368, identified as cholesta-3,5-diene (see Fig. 5.3.b). Significant amounts of a number of compounds of proteinaceous origin were also present. The 2,5-diketopiperazine derivative proline-glycine (pro-gly) (m/z 70, 83, 111, M^+ 154) was identified as a major component eluting just prior to the $C_{14:0}$ fatty acid. Two 2,5-diketopiperazine derivatives of proline-alanine (pro-ala) (m/z 70, 97, 125, M^+ 168) were also identified, as were other diketopiperazine derivatives at lower abundance. Other compounds noted were octadecanenitrile ($[M-15]^+$ 250) characterised by ions of m/z 110 and 124, and $C_{16:0}$ (M^+ 255), $C_{17:0}$ (M^+ 269), $C_{18:1}$ (M^+ 281) and $C_{18:0}$ (M^+ 283) amides characterised by ions of m/z 59 and 72. *n*-Nonanal was present as a minor component. Typical lipid pyrolysis products (e.g. alkenes, alkanes, ketones etc.) were absent. Yet products normally associated with the pyrolysis of proteins (Stankiewicz et al. 1996, p.1747; Stankiewicz et al. 1997, p.1884; Munson & Fetterolf 1987, p.15) were seen, suggesting that they may have been formed at relatively low temperatures during the TD at 290/310°C. Their absence from the solvent extracts (see below) suggests that they are probably not present in their free form in the ancient tissues.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.2a, inset. The pyrogram is dominated by alkene/alkane doublets (m/z 55/57; C_6 to C_{24}), with the *n*- C_{15} homologue most abundant. Methyl ketones (C_{10} , C_{11} and C_{16} to C_{19}) characterised by mass chromatograms m/z 58 and 71 and more unusually ethyl (3-) ketones (m/z 57 and 72; C_{18} and C_{19}) were identified. Saturated ($C_{14:0}$ to $C_{18:0}$) and unsaturated ($C_{18:1}$) nitriles were also present as major components, with the $C_{16:0}$ and $C_{18:0}$ alkyl amides present as relatively

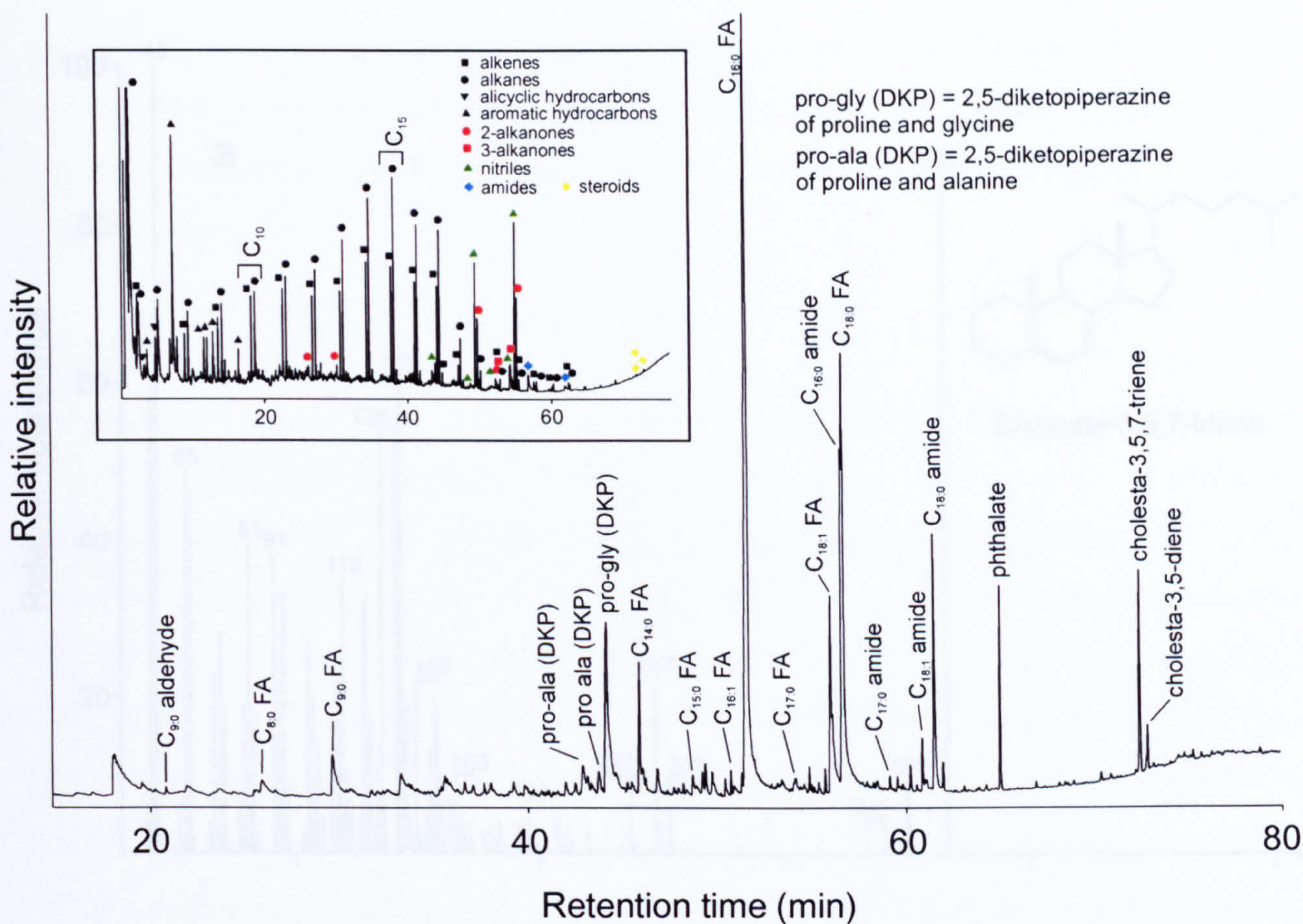


Figure 5.2a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin/tissue'/bandaging from the Middle Kingdom male adult 'Khnumnakht', XIIth dynasty (c. 1900 B.C.).

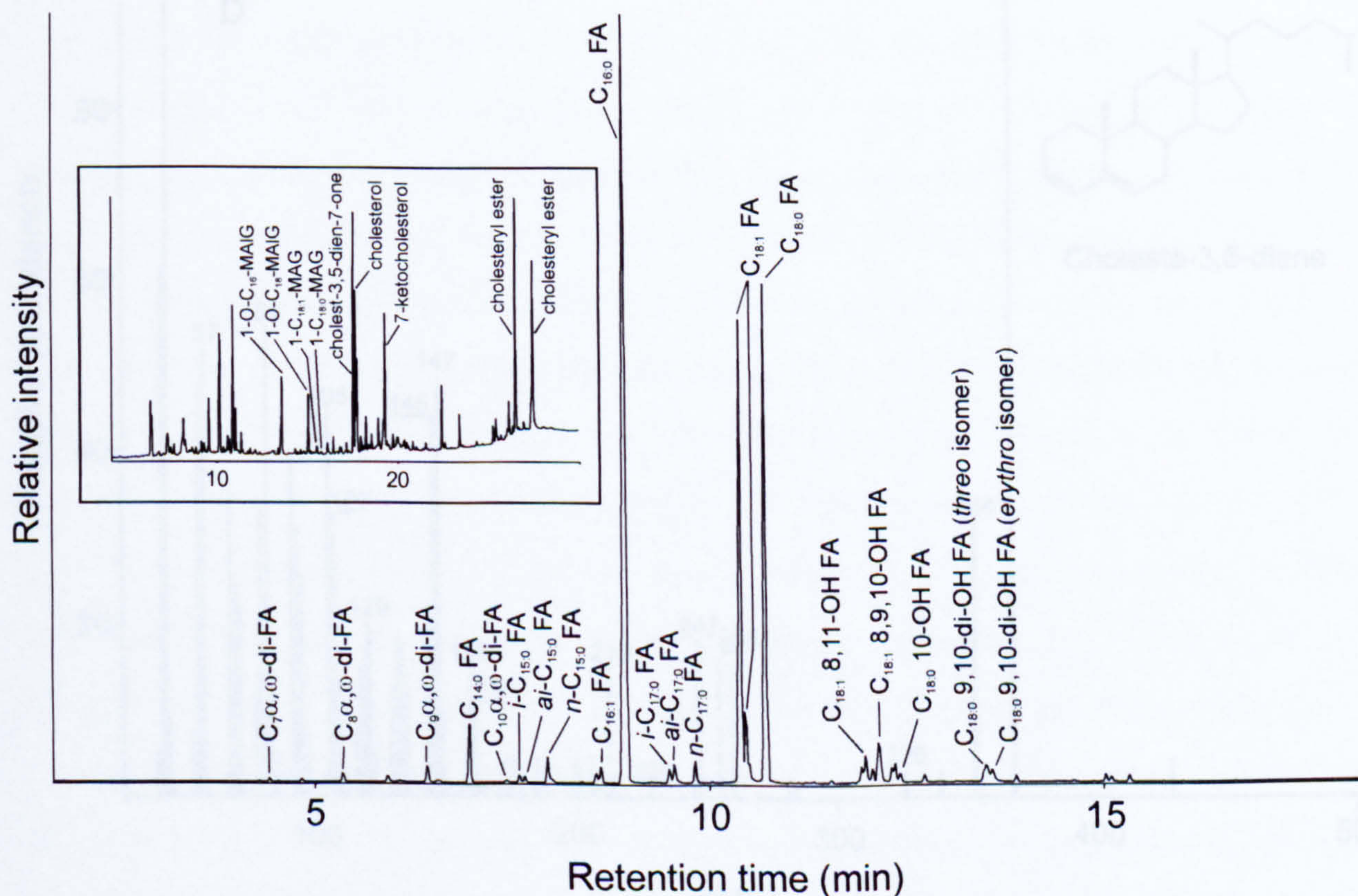


Figure 5.2b Total ion chromatograms from the GC/MS analyses of the acid fraction, and (inset) neutral fraction of 'resin/tissue'/bandaging from the Middle Kingdom male adult 'Khnumnakht', XIIth dynasty (c. 1900 B.C.).

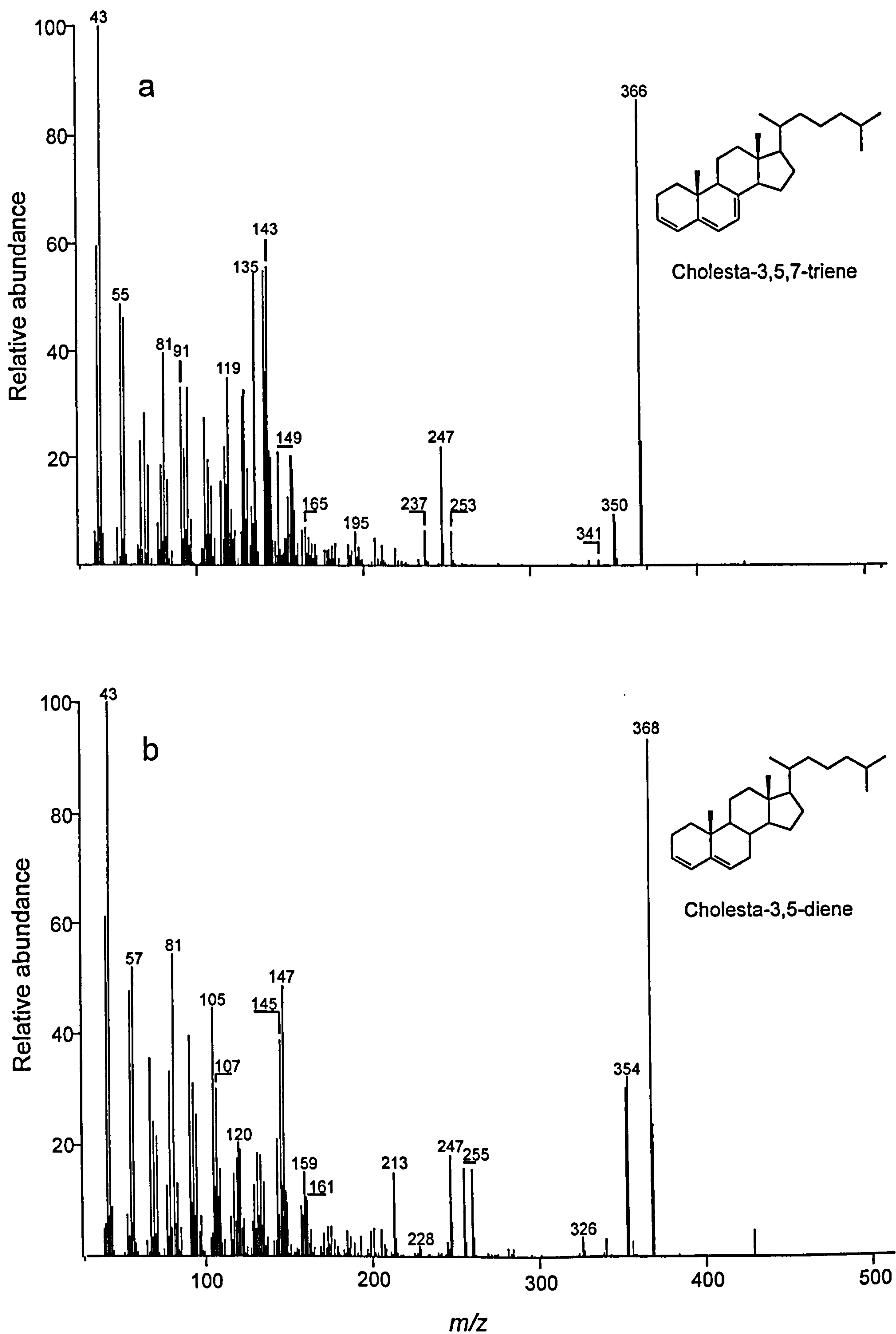


Figure 5.3 Mass spectra of the two steroidal compounds detected in the 'resin/tissue' bandaging from the male adult 'Khnumnakht' after thermal desorption at 310°C for 10s; (a) cholesta-3,5,7-triene and (b) cholesta-3,5-diene

minor components and benzene and cyclopentane derivatives also detected. Cholest-(?)-ene (M^+ 370), cholesta-3,5,7-triene and cholesta-3,5-diene were also observed, as was a pyrrole. The pyrolysate constitutes a significant portion of the sample (i.e. cf. TD), indicating the presence of abundant bound biomarkers and pointing to a significant degree of polymerisation.

GC/MS – Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.2b. A range of carboxylic acids was identified, the chromatogram being dominated by a series of monocarboxylic acids. This included saturated straight chain acids in the range C_{12} to C_{24} carbon number range (with the exception of C_{21}), the saturated *iso*- and *anteiso*-methyl branched acids in the C_{15} to C_{17} range, and unsaturated acids including $C_{16:1}$, $C_{18:1}$, $C_{20:1}$ and $C_{24:1}$. The profile of the monocarboxylic acids, dominated by the $C_{16:0}$, $C_{18:0}$ and $C_{18:1}$ components, was comparable with the TD profile obtained for this sample. A series of α,ω -dicarboxylic acids in the C_7 to C_{11} carbon number range were present as relatively minor components together with moderate amounts of monohydroxy and dihydroxy carboxylic acids.

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.2b, inset. The neutral fraction was dominated by cholesteryl esters and several related steroidal compounds, with monoglyceryl ethers making a significant, albeit minor, contribution. The major free steroid identified was cholesta-3,5-dien-7-one with cholesterol and 3-hydroxycholest-5-en-7-one (7-ketocholesterol) also abundant. At longer retention times (26.5 and 27.4 min) two cholesteryl esters were present, again as major components. These would have been too involatile to successfully elute from the column utilised in the TD-GC/MS analyses, though it is anticipated that they would undergo a 1,2-elimination upon TD at 290/310°C to give the fatty acid and cholesta-3,5-diene (with a lesser amount of cholesta-3,5,7-triene, *via* dehydrogenation). Also significant were the $C_{16:0}$ and $C_{18:0}$ 1-*O*-monoalkylglyceryl ethers (as their bis-TMS ethers), although the $C_{18:0}$ and $C_{18:1}$ 1-monoacylglycerols were detected only as minor components and other 1-monoacylglycerols were absent, indicating almost complete hydrolysis of the original acyl lipids that would have dominated the tissues at the time of death (if indeed they do originate from the body). The neutral fraction constitutes a significant proportion of the total lipid extracts of the sample. The steroids and sterol esters are present in similar abundance to the $C_{15:0}$ and

C_{17:0} fatty acids, although the C_{16:0}, C_{18:0} and C_{18:1} components constituted the bulk of the extractable lipid present in the sample.

5.4.3 Female Adult, XVIIth dynasty (c.1650-1550 BC), Second Intermediate Period, Qurna/Thebes (1909.527)

5.4.3.1 'Resin/tissue' from head of right tibia [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.4a. A series of monocarboxylic acids (C₁₄ to C₁₈) were detected, the major components being C_{16:0}, C_{18:1} and C_{18:0} in decreasing order of abundance. Two C_{18:2} fatty acids were also identified as significant constituents. As observed in Khnumnakht (5.4.2 above), the cholesterol derivatives cholesta-3,5,7-triene and cholesta-3,5-diene were significant in their abundance, as were the 2,5-diketopiperazine derivatives pro-ala (two isomers) and pro-gly, the latter component comparable in abundance to the two C_{18:2} fatty acids. Although only present as trace components the two diterpenoids methyl dehydroabietate (characterised by a base peak of m/z 239) and methyl 7-oxodehydroabietate (characterised by a base peak of m/z 253 and M^+ 328) were also tentatively identified, as were the C₂₇ and C₃₁ *n*-alkanes.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.4a, inset. The C_{16:0}, C_{18:0}, C_{18:1} and C_{18:2} fatty acids, the steroidal compounds cholesta-3,5,7-triene and cholesta-3,5-diene, and the 2,5-diketopiperazines derivatives pro-ala (two isomers) and pro-gly were identified. The relative abundance of the fatty acids *cf.* TD suggests that these fatty acids derive from polymeric acyl lipids rather than from free components trapped in the sample matrix (see Chapter 4). In contrast, steroidal compounds are likely, in part at least, to be the free components too involatile to reach the GC column after volatilisation at 310°C (Buckley & Evershed, unpublished data).

GC/MS – Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.4b. The major components were monocarboxylic acids (C₁₄ to C₂₄), with C_{16:0}, C_{18:0} and C_{18:1} predominating. The distribution of monocarboxylic acids was similar to that obtained using TD (see Figure 5.4). Notably the relatively labile C_{18:1} fatty acid, was the second

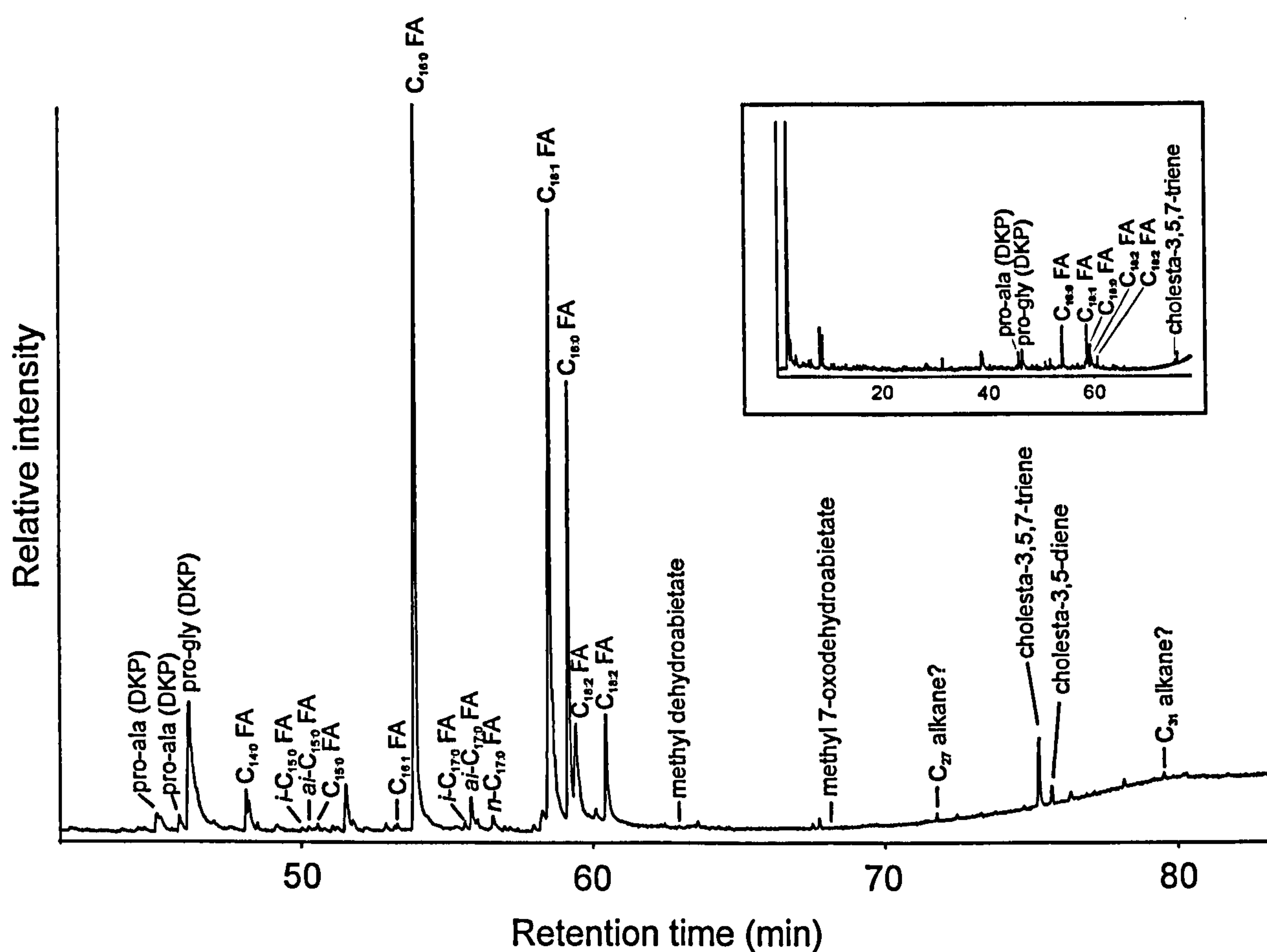


Figure 5.4a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin/tissue' from the head of the right tibia [1] of the Second Intermediate Period female adult, XVIIth dynasty (c. 1650-1550 B.C.).

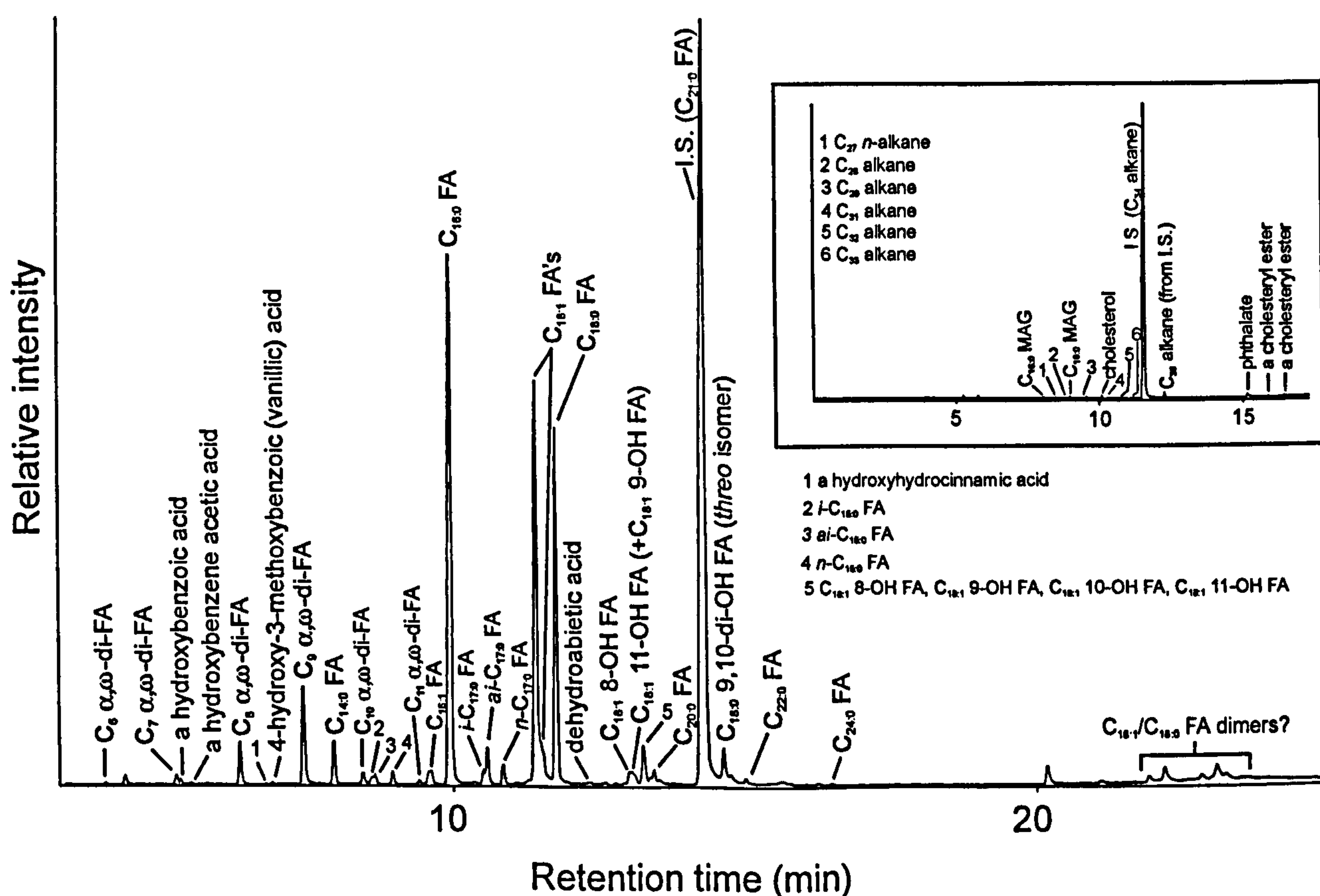


Figure 5.4b Total ion chromatograms from the GC/MS analyses of the acid fraction, and (inset) neutral fraction of 'resin/tissue' from the head of the right tibia [1] of the Second Intermediate Period female adult, XVIIth dynasty (c. 1650-1550 B.C.).

most abundant component (after palmitic acid) in the acid fraction. Branched chain fatty acids ($C_{15:0}$ and $C_{17:0}$) were identified as minor components, whilst present in significant abundance were α,ω -dicarboxylic acids (C_6 to C_{11}), with C_8 and C_9 predominating. Also significant were the mono- and dihydroxy carboxylic acids, with the $C_{18:1}$ monohydroxy acids in similar abundance to the $C_{18:0}$ 9,10-dihydroxy fatty acids, the *threo* isomer predominating in the latter. Fatty acid dimers (linked mid-chain; Koster et al. 1998, p.159-169) were also tentatively identified eluting between 21.5 and 23.8 min. A number of aromatic acids, namely, (?) -hydroxybenzoic, (?) -hydroxybenzeneacetic, (?) -hydroxyhydrocinnamic and 4-hydroxy-3-methoxybenzoic (vanillic) acids were also observed, albeit as minor components. The most interesting of the compounds identified, albeit as a trace component, was dehydroabietic acid (as its TMS ester) characterised by M^+ 372, $[M-15]^+$ 357, and a base peak of m/z 239. The presence of the methyl ester of this acid in the TD-GC/MS, which is formed in the probe at relatively low temperatures (290/310°C), suggests that it is present in the sample rather than due to contamination.

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.4b, inset. Minor quantities of the $C_{16:0}$ and $C_{18:0}$ 1-monoacylglycerols (as the bis-TMS ethers) were observed along with C_{27} to C_{33} alkanes with no odd-over-even preference. Cholesterol (as its TMS ether) plus two cholesteryl esters were also identified as minor components. The neutral components constituted relatively little of the solvent extractable material (4%), the internal standard (C_{34} *n*-alkane) being by far the major compound present (see Figure 5.4b, inset).

5.4.3.2. Textile material/‘fatty matter’ from mass of cloth [2].

TD-GC/MS

The results of the TD-GC/MS analysis displayed a series of monocarboxylic acids (C_{14} to C_{18}) with the $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ predominating in increasing order of abundance. The steroidal compounds cholesta-3,5,7-triene and cholesta-3,5-diene were detected in similar relative abundance to the previous sample, with C_{27} to C_{31} alkanes present as minor constituents. Yet unlike sample 5.4.3.1, no diunsaturated fatty acids or 2,5-diketopiperazines indicative of a proteinaceous origin were observed. Again methyl dehydroabietate was tentatively identified as a trace component.

Py-GC/MS

In the Py-GC/MS analysis, C_{16:0}, C_{18:0}, C_{18:1} and C_{18:2} fatty acids, cholesta-3,5,7-triene and cholesta-3,5-diene were present in the same relative abundance as the previous sample from the XVIIth dynasty female adult. The presence of these compounds as relatively minor components compared to those of the TD makes their origin difficult to ascertain despite their similarity with the previous sample. Also observed as a major component of the pyrolysate was levoglucosan, with significant amounts of furan and pyran derivatives. Given the origin of the sample these pyrolysis products are likely to derive from the cellulose making up the bulk of the textile present.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS revealed a series of monocarboxylic acids (C₁₄ to C₂₄). These dominated the chromatogram, their distribution being similar to that obtained using TD. The major component was C_{18:0} fatty acid, although the C_{18:1} FA was not quite as abundant with an increased proportion of the C_{16:0} fatty acid. The C_{15:0} and C_{17:0} branched chain fatty acids were in similar relative abundance to the previous sample from this mummy. Present in significant abundance were α,ω -dicarboxylic acids (C₆-C₁₀), with C₈ and C₉ predominating. Also significant were the mono- and dihydroxy carboxylic acids, with the C_{18:1} monohydroxy acids in similar abundance to the C_{18:0} 9,10-dihydroxy fatty acids, with the *threo* isomer predominating in the latter. Fatty acid dimers were also tentatively identified. Unlike the previous sample no hydroxy aromatic acids were observed, but the diterpenoid dehydroabietic acid (as its TMS ester) was identified as a trace component, its presence in the sample being corroborated by the identification of the thermolytically derived methyl ester of the acid in the TD-GC/MS.

GC/MS-Neutral fraction

The results for the neutral fraction analysed by GC/MS revealed moderate quantities of the C_{16:0} and C_{18:0} 1-monoacylglycerols (as their bis-TMS ethers), and significant amounts of C₂₇-C₃₃ alkanes with no odd-over-even preference. Cholesterol (as its TMS ether) and two cholesteryl esters were also identified as the major components of the neutral fraction. However, like the previous sample for this mummy, the neutral fraction constituted only a small proportion of the total extract (2%).

5.4.4 Child(?), XVIIth dynasty (c.1650-1550 BC), Second Intermediate Period, Qurna/Thebes (1909.527)

5.4.4.1 Unspecified bone and cartilage [2].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.5a. A series of monocarboxylic acids (C_{14} to C_{18}) was detected, the major components being $C_{16:0}$, $C_{18:0}$ and $C_{18:1}$ in decreasing order of abundance. Interestingly, two $C_{18:2}$ fatty acids were also identified as significant constituents. The cholesterol derivatives, cholesta-3,5,7-triene and cholesta-3,5-diene were present in significant abundance, as were the 2,5-diketopiperazines derivatives pro-ala (two isomers) and pro-gly, the latter being the fourth most abundant compound after the $C_{16:0}$, $C_{18:0}$ and $C_{18:1}$ fatty acids. C_{27} to C_{31} alkanes with no odd-over-even predominance were identified as minor constituents.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.5a, inset. The $C_{16:0}$, $C_{18:0}$, $C_{18:1}$ and $C_{18:2}$ fatty acids, the steroidal compounds cholesta-3,5,7-triene and cholesta-3,5-diene and the 2,5-diketopiperazines derivatives pro-ala (two isomers) and pro-gly were identified. The relative abundance of the fatty acids *cf.* TD suggests that these fatty acids derive from polymeric acyl lipids, rather than from free components trapped in the sample matrix (see Chapter 4). In contrast, steroidal compounds are likely, in part at least, to be the free components too involatile to be transferred to the GC column after volatilisation at 310°C.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.5b. The major components were monocarboxylic acids (C_{14} to C_{24}), with $C_{16:0}$, $C_{18:0}$ and $C_{18:1}$ predominating. The distribution of monocarboxylic acids was similar to that obtained using TD (see Figure 5.5). As was observed in the samples from the XVIIth dynasty female adult, the $C_{18:1}$ fatty acid was one of the three major constituents. Branched chain fatty acids ($C_{15:0}$ and $C_{17:0}$) were identified as minor components. Present in significant abundance were α,ω -dicarboxylic acids (C_6 to C_{11}), with C_8 and C_9 predominating. Also significant were the mono- and dihydroxy carboxylic acids, with the $C_{18:1}$ monohydroxy acids in similar abundance to the $C_{18:0}$ 9,10-dihydroxy fatty acids, the *threo* isomer predominating in the latter. Fatty acid dimers were also tentatively identified, eluting

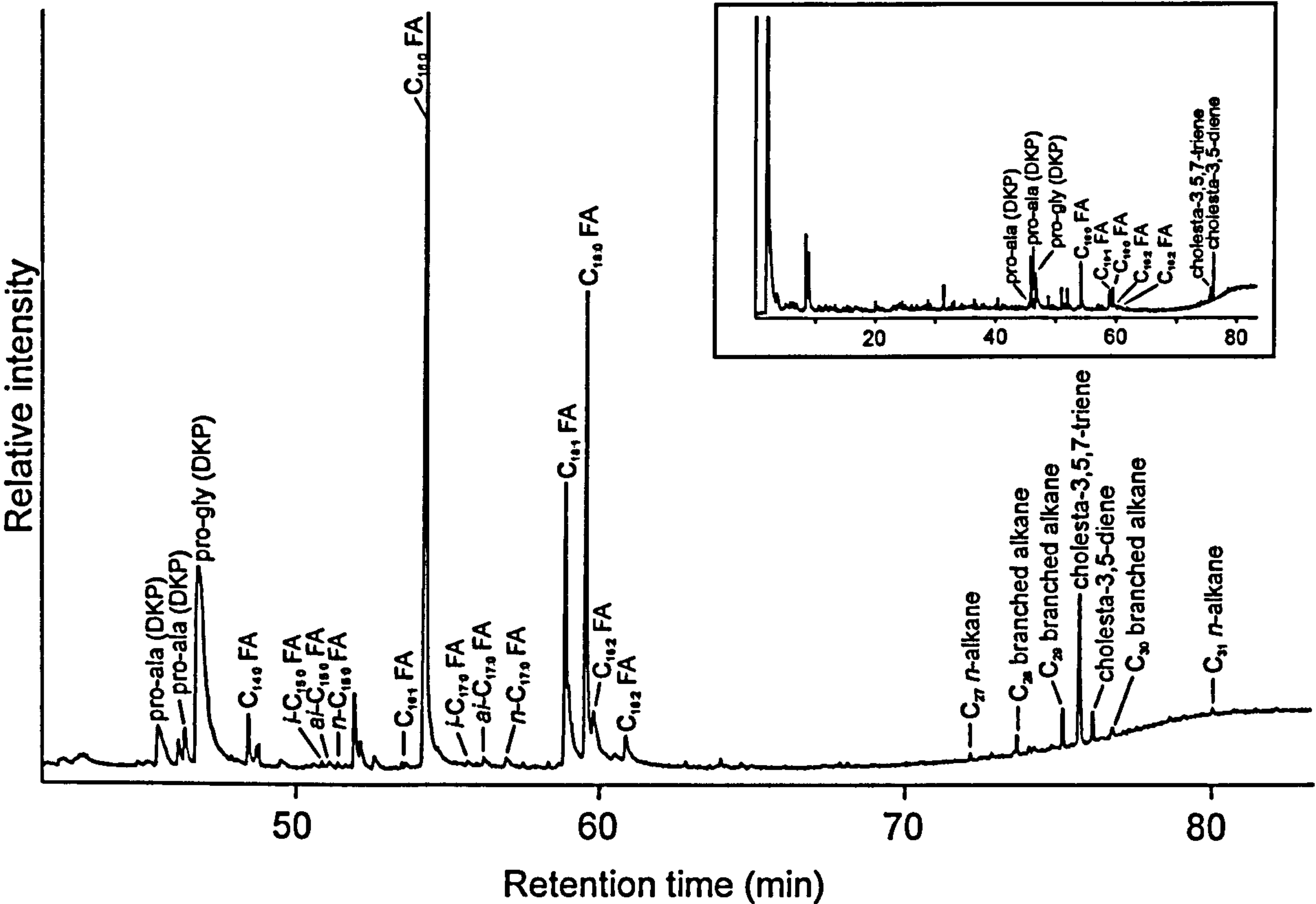


Figure 5.5a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of unspecified bone and cartilage [2] from the Second Intermediate Period Child (sex unknown), XVIIth dynasty (c. 1650-1550 B.C.).

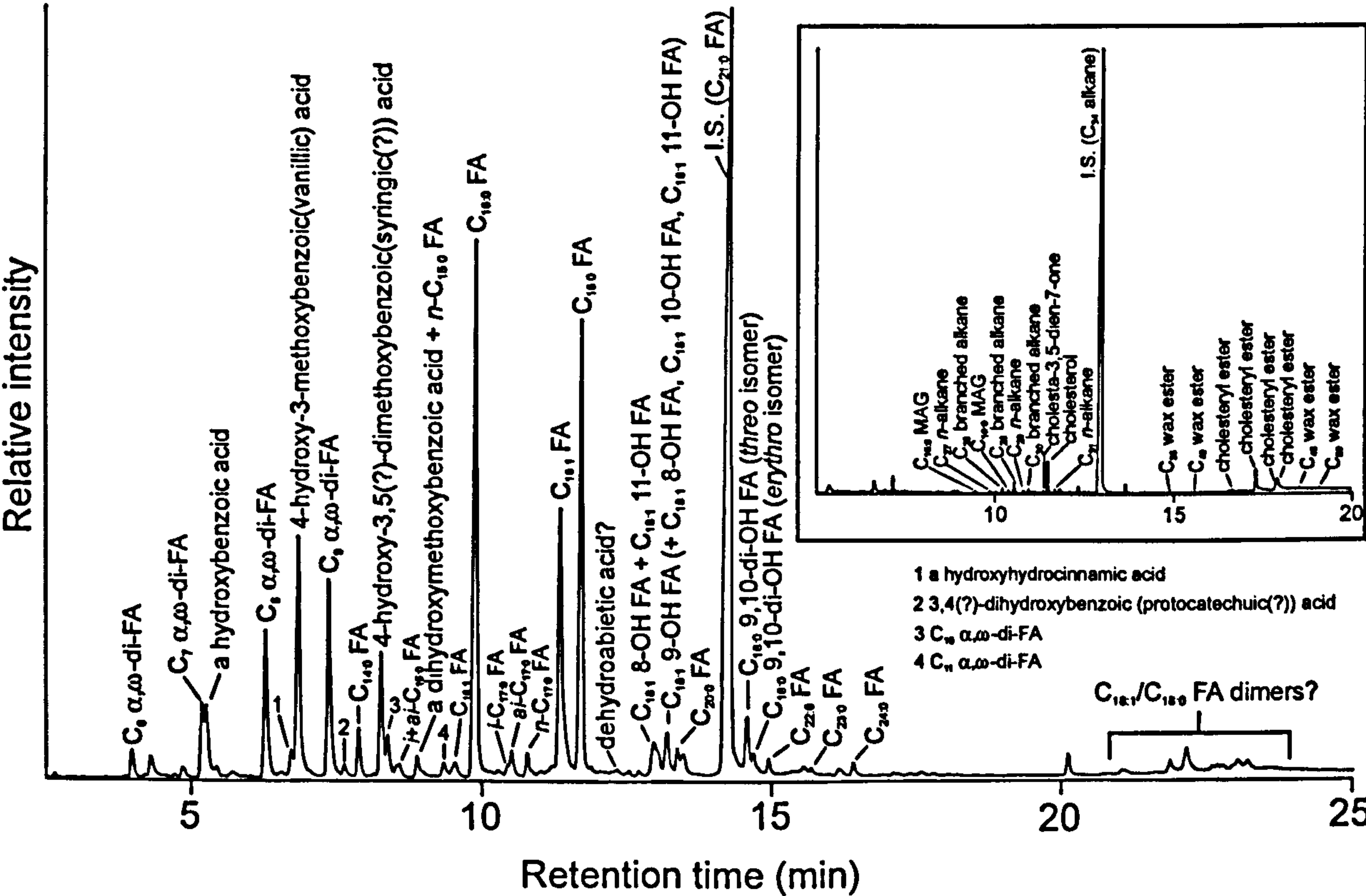


Figure 5.5b Total ion chromatograms from the GC/MS analyses of the acid fraction, and (inset) neutral fraction of unspecified bone and cartilage [2] from the Second Intermediate Period Child (sex unknown), XVIIth dynasty (c. 1650-1550 B.C.).

between 20.8 and 23.8 min. Significant components in this sample were a number of hydroxy aromatic acids (as their TMS derivatives) identified as 4-hydroxy-3-methoxybenzoic (vanillic), 4-hydroxy-3,5(?) -dimethoxybenzoic (syringic), 4(?) -hydroxybenzoic, 4(?) -hydroxyhydrocinnamic, ?,?-dihydroxy-?-methoxybenzoic and 3,4-dihydroxybenzoic (protocatechuic) acids in decreasing order of abundance. They were identified by their mass spectra, shown in Figure 5.6 (a-f). These compounds were not seen in the TD-GC/MS presumably on account of their highly polar nature. Although barely detectable, dehydroabietic acid was tentatively identified (coeluting with a C_{19:0} fatty acid) as a trace component, characterised by the ions M⁺ 372, [M-15]⁺ 357, and base peak *m/z* 239. However, the absence of both a good mass spectrum, and its methyl ester in the TD analysis make its significance uncertain.

GC/MS Neutral-fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.5b, inset. Minor quantities of the C_{16:0} and C_{18:0} 1-monoglycerols (as their bis-TMS ethers) were detected, as were cholesterol (as its TMS ether), cholesta-3,5-dien-7-one and four cholesteryl esters. Alkanes (C₂₇ to C₃₂) with no odd-over-even preference were observed along with a number of wax esters (C₃₈, C₄₀, C₄₈ and C₅₀), both compound classes present as significant constituents of the neutral fraction. Wax esters, in the C₄₀ to C₅₀ carbon number range, appeared to be coeluting with the relatively more dominant cholesteryl esters. The neutral fraction constituted a significant, but relatively small, proportion of the total extract (10%).

5.4.4.2 Stained wrapping [3]

TD-GC/MS

The results of the TD-GC/MS analysis revealed the C_{16:0} and C_{18:0} as the two major components with very little free ('unbound') material present. This is consistent with the nature of the sample, i.e. linen wrapping.

Py-GC/MS

The results of the Py-GC/MS revealed that the major component of the pyrolysate (excluding CO₂) was levoglucosan, in addition to significant amounts of furan and pyran derivatives, no doubt deriving from the linen wrappings.

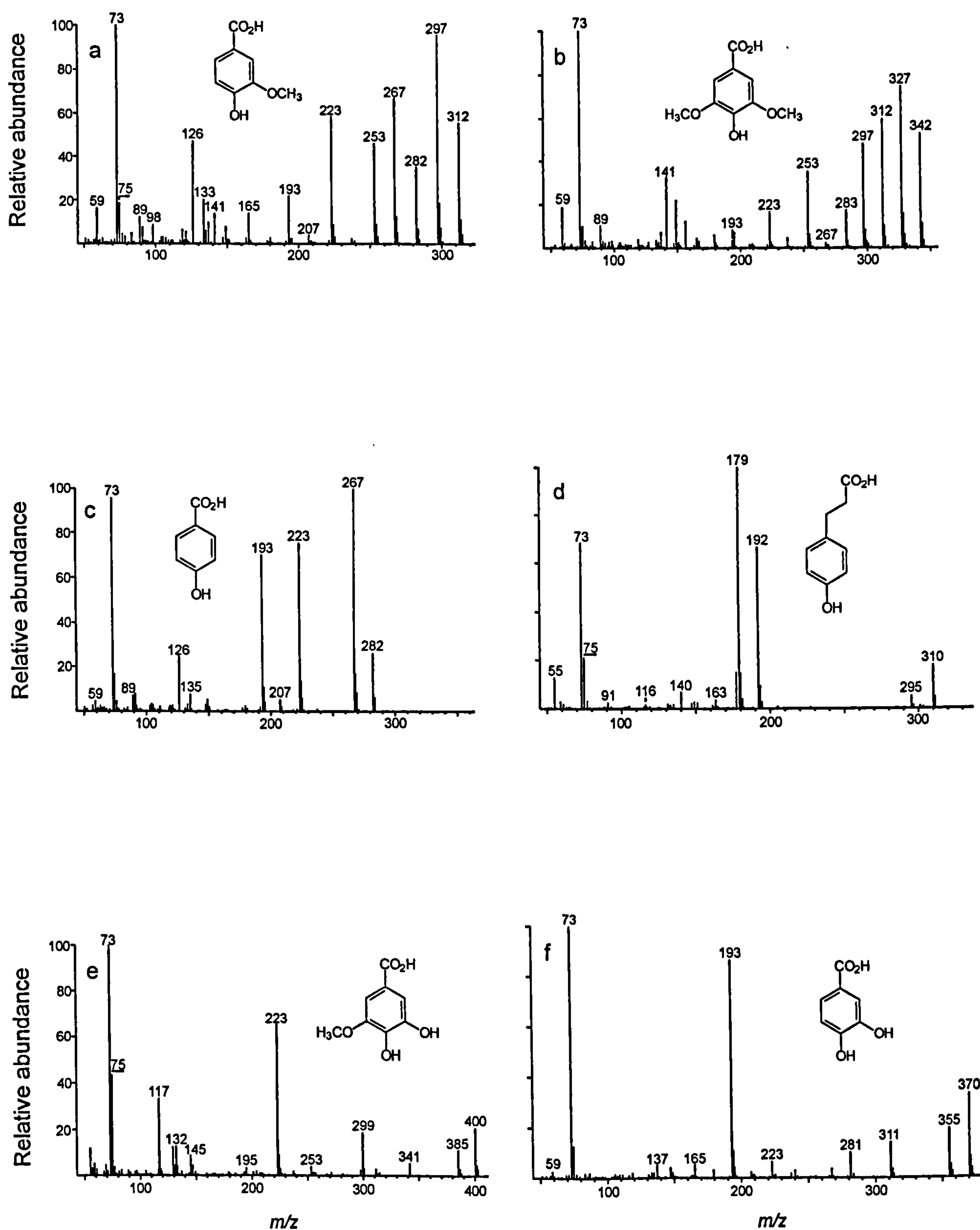


Figure 5.6 Mass spectra of the hydroxy aromatic acids (as their TMS derivatives) identified in the acid fraction of the unspecified bone and cartilage [2] from the Second Intermediate Period Child (sex unknown), XVIIth dynasty (c. 1650-1550 B.C.).

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS displayed a series of monocarboxylic acids (C_{14} to C_{24}), with $C_{18:0}$ and $C_{16:0}$ the major compounds in decreasing order of abundance, and were the two main components in the acid fraction. The $C_{18:1}$ fatty acid was present only as a minor component, unlike the bone and cartilage sample from the child(?) mummy. Yet, the increased abundance of the α,ω -dicarboxylic acids (C_6 - C_{11}) present in the stained wrappings *cf.* the previous sample suggests that the $C_{18:1}$ fatty acid would originally have been present in higher abundance. In addition to the diacids, $C_{18:1}$ monohydroxy- and $C_{18:0}$ 9,10-dihydroxy carboxylic acids were present as minor constituents, the *threo* isomer predominating in the latter. Vanillic acid was identified as a minor component, but the additional aromatic acids noted in the previous sample were not observed in this case. Although only very tentatively identified in the aforementioned sample taken from the XVIIth dynasty child(?), dehydroabietic acid was identified as a minor component, clearly characterised by its mass spectrum (M^+ 372, $[M-15]^+$ 357, and base peak m/z 239). Due to the presence of very few free components in the TD analysis, a meaningful comparison was not possible in this instance.

GC/MS-Neutral fraction

The results for the neutral fraction analysed by GC/MS revealed very minor quantities of the $C_{16:0}$ and $C_{18:0}$ 1-monoacylglycerols and trace quantities of alkanes (C_{27} - C_{32}) with no odd-over-even preference. Cholesterol and its derivatives were also present as trace or minor components, the neutral fraction as a whole constituting very little of the total solvent extractable material (1%).

5.4.5 Head, XVIIIth-XXth dynasty (c.1550-1069 BC), New Kingdom, Thebes (1976.159.267)

5.4.5.1 'Resin/skin' beneath right eye/orbit [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.7a. A series of monocarboxylic acids (C_{14} to C_{18}) were detected, the major components being $C_{16:0}$, $C_{18:0}$ and $C_{14:0}$ in decreasing order of abundance. The cholesterol derivative cholesta-3,5,7-triene was present as a significant component, with cholesta-3,5-diene identified in much lower abundance. Also present as minor components were the 2,5-diketopiperazine derivatives pro-gly and pro-ala (two isomers) and the diterpenoid methyl 7-oxodehydroabietic acid.

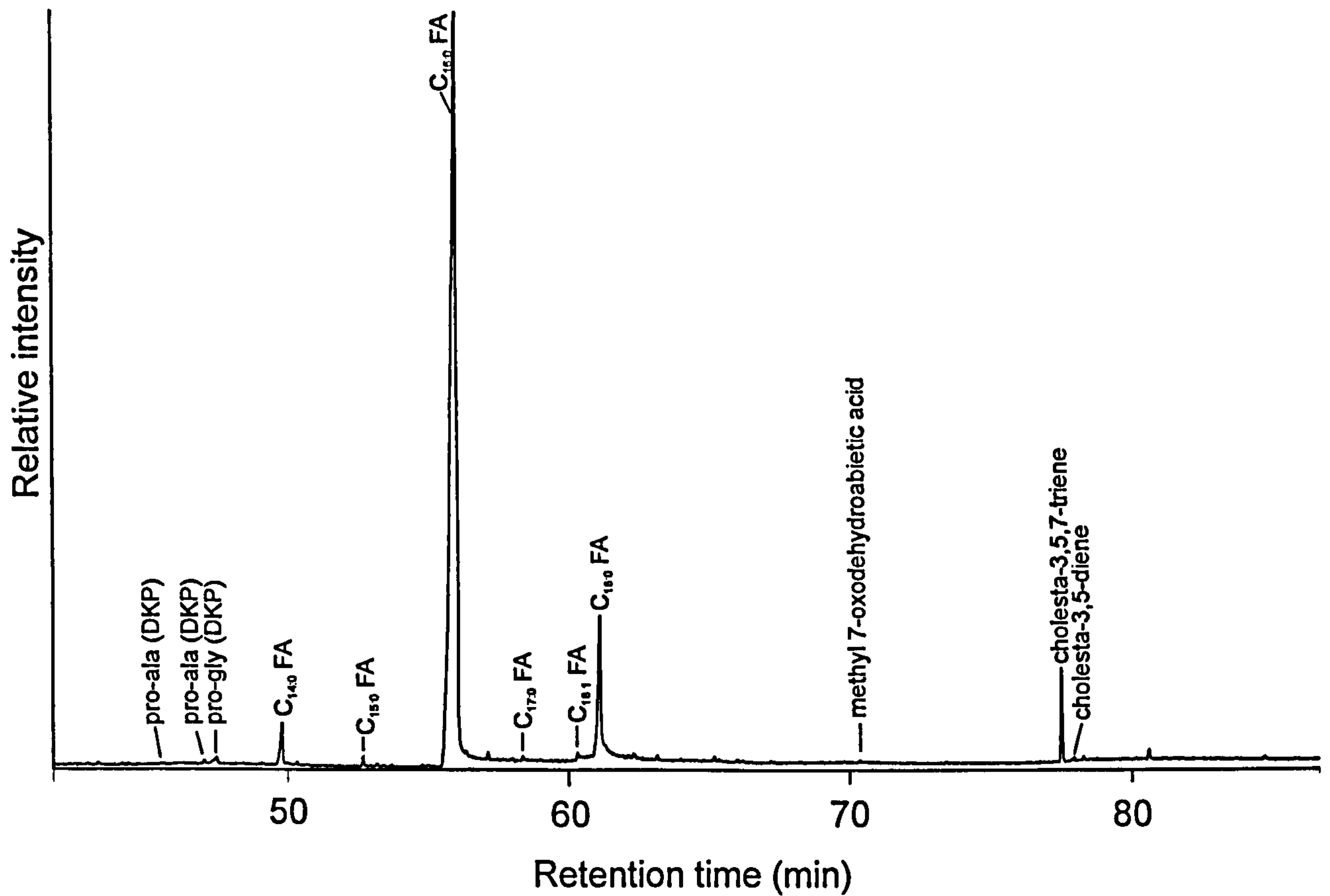


Figure 5.7a Total ion chromatogram of the thermal desorption profile (310°C/10s) of 'resin/skin' from beneath the right eye/orbit [1] of the New Kingdom head, XVIIIth–XXth dynasty (c. 1550-1069 B.C.).

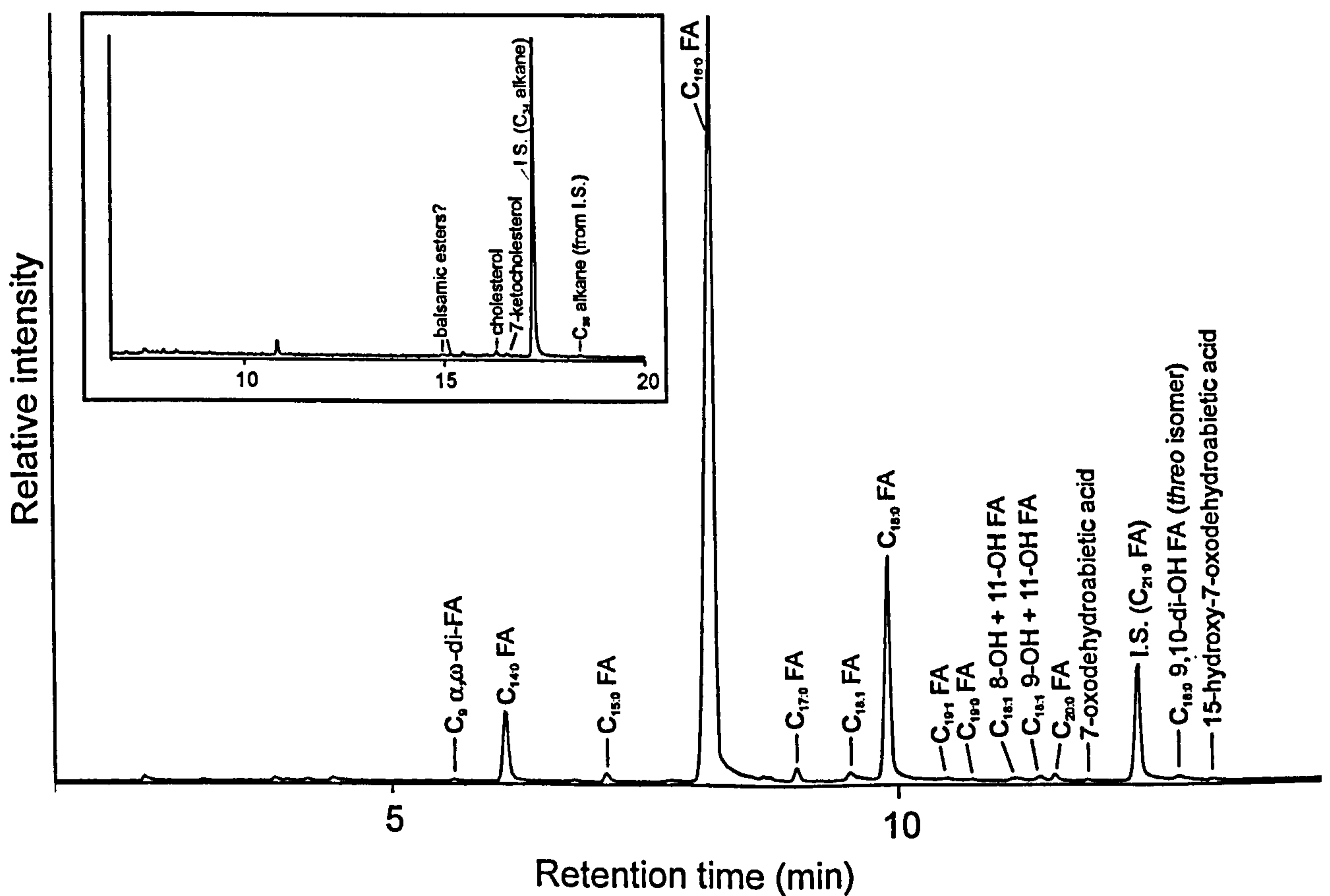


Figure 5.7b Total ion chromatograms from the GC/MS analyses of the acid fraction, and (inset) neutral fraction of 'resin/skin' from beneath the right eye/orbit [1] of the New Kingdom head, XVIIIth–XXth dynasty (c. 1550-1069 B.C.).

Py-GC/MS

The results of the Py-GC/MS analysis revealed no significant pyrolysate, indicating that no bound biomarkers of any appreciable abundance were present in the sample (highly polar material could have been present, although this would not have eluted from the column, or indeed volatilised from the probe).

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.7b. The major components were monocarboxylic acids (C_{14} to C_{20}), with $C_{16:0}$, $C_{18:0}$ and $C_{14:0}$ predominating in decreasing order of abundance. The distribution of monocarboxylic acids was similar to that obtained using TD (see Fig. 5.7). The $C_{18:1}$ fatty acid which was abundant in the previous two mummies was present only as a minor constituent. Also observed as a minor component was azelaic acid (C_9 α,ω -dicarboxylic acid) although the C_6 - C_8 diacids were not detected). The mono- and dihydroxy carboxylic acids were again only present as minor components, the $C_{18:1}$ -OH and the $C_{18:0}$ 9,10-di-OH fatty acids in similar abundance. Two oxidised diterpenoids were also observed as minor constituents. 7-Oxodehydroabietic acid as its TMS ester was characterised by the presence of m/z 73, 268 and base peak 253, and 15-hydroxy-7-oxodehydroabietic acid as the free hydroxy, TMS ester (steric hindrance preventing the trimethylsilylation of the hydroxyl group) by m/z 73, 269, 284 and $[M-15]^+$ 387. The former of these was also observed in the TD-GC/MS as its thermolytically derived methyl ester.

GC/MS Neutral-fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.7b, inset. Cholesterol (as its TMS ether) and 7-ketocholesterol (as its TMS ether) were present as minor components, as were two tentatively assigned balsamic esters. The neutral components constituted very little of the total solvent extractable material (1%).

5.4.5.2 'Skin/resin' from back of cranium [3]

TD-GC/MS

The results of the TD-GC/MS analysis displayed a series of monocarboxylic acids (C_{14} to C_{18}) with $C_{16:0}$ and $C_{18:0}$ predominating. The cholesterol derivative cholesta-3,5,7-triene was a significant component, with cholesta-3,5-diene identified as a minor component. Present in significant quantities were the 2,5-diketopiperazine (DKP) derivatives pro-ala (two isomers) and pro-gly, the latter DKP the second most abundant compound in the TD

after the C_{16:0} fatty acid. Also present as a minor but important component was methyl 7-oxodehydroabietate. With the exception of the abundant DKP derivatives present in this sample, the TD profiles of the two samples from the New Kingdom mummified head are notably similar.

Py-GC/MS

The Py-GC/MS showed that DKP derivatives were again abundant, with an absence of C_{16:0}, C_{18:0} and C_{18:1} monocarboxylic acids, suggesting that the fatty acids present in the sample are free components rather than being bound within a polymeric matrix.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS revealed a series of monocarboxylic acids as the major acidic components, with C_{16:0} and C_{18:0} predominating. The distribution of monocarboxylic acids was similar to that obtained using TD. Again the C_{18:1} fatty acid was only present as a minor component. Unlike the previous sample 5.4.5.1, α,ω -dicarboxylic acids (C₈ to C₁₀) were observed as significant components which suggests a greater degree of oxidation in this sample. The mono- and dihydroxy carboxylic acids were also present in much greater abundance than in sample 5.4.5.1, which is again consistent with it having undergone more extensive oxidative degradation. These mono- and dihydroxy fatty acids were in similar abundance to the C_{14:0} fatty acid. Vanillic acid was also identified as a significant component. Three diterpenoid acids were also detected, namely dehydroabietic, 7-oxodehydroabietic and 15-hydroxy-7-oxodehydroabietic acid, the former only present as a trace component. The presence of diterpenoids was also confirmed by the methyl 7-oxodehydroabietic detected in the TD-GC/MS analysis.

GC/MS Neutral-fraction

The results for the neutral fraction analysed by GC/MS revealed minor quantities of the C_{16:0} and C_{18:0} 1-monoacyl-glycerols with C₃₀ to C₃₂ alkanes present in similar abundance. Cholesterol and 7-ketocholesterol were also identified as minor components, as was levoglucosan (as its TMS derivative). A methyl ester of methoxy- α -hydroxybenzeneacetic acid (as its TMS ether) was also tentatively identified along with two other aromatic (balsamic) esters. The neutral components constituted a significant, but relatively small, proportion of the total solvent extractable material (10%).

5.4.6 Female Adult, XXIst–XXVth dynasty (c.1069-664 BC), Third Intermediate Period, Thebes(?) (EA.74303)

5.4.6.1 'Resin' from thoracic cavity [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.8a. A series of monocarboxylic acids (C₈ to C₁₈) were detected, the major component being C_{16:0}, this acid dominating the TD profile. The C₉ fatty acid was also significant, as were the C_{16:0} γ - and δ -lactones. The diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate were observed as significant components together with a complex suite of decarboxylated 7-oxo- diterpenoids the major components of which being identified as 7-oxo-18-norabieta-3,5,8,11,13-pentaene (m/z 209, 238, 251 (base peak) and M⁺ 266) and 7-oxo-18-norabieta-3,5,8,11,13,15-hexaene (m/z 207, 236, 249 (base peak) and M⁺ 264). Retene (m/z 189, 204, 219 (base peak) and M⁺ 234) and 15-dehydreretene (m/z 192, 202, 217 and M⁺ 232 (base peak) were also identified. The structures of these diterpenoids are shown in Figure 5.9 with their corresponding mass spectra. Also significant components were a series of *n*-alkanes, C₂₅ to C₃₃, with an odd-over-even preference.

Py-GC/MS

The results of the Py-GC/MS are shown in Figure 5.8a, inset. The pyrogram is dominated by alkene/alkane doublets (m/z 55/57) (C₈ to C₂₄) maximising *n*-C₉ to C₁₁. Free fatty acids (C_{7:0} to C_{9:0}) were also observed along with C_{16:0} and C_{18:0}, the abundances of these acids indicating their origin from a bound fraction of this sample, either as part of the polymeric lipid or as constituents trapped in the sample matrix. Retene and a higher homologue of retene were significant components along with similar abundances of decarboxylated 7-oxo- diterpenoids, and minor quantities of methyl 7-oxo-dehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.8b. A series of monocarboxylic acids (C₉ to C₃₀) with C_{16:0}, C_{18:0} and C_{14:0} the major components in decreasing order of abundance. Also present in appreciable quantities were α,ω -dicarboxylic acids (C₄ to C₁₄) with C₈ and C₉ predominating. Minor amounts of the C_{18:0} 9,10-dihydroxy acids (*threo* and *erythro* isomers) were also detected along with the

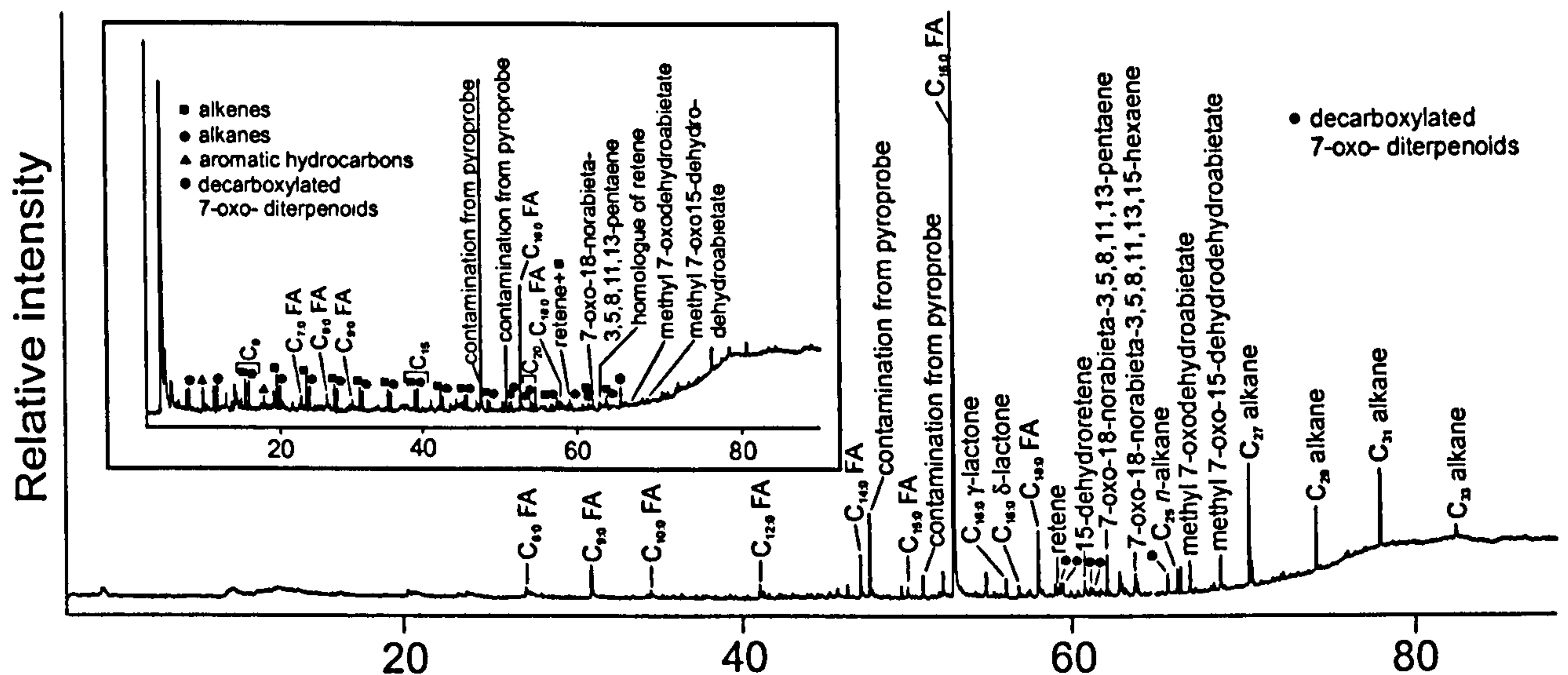


Figure 5.8a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin' from the thoracic cavity [1] of the Third Intermediate Period female adult, XXIst-XXVth dynasty (c. 1069-664 B.C.).

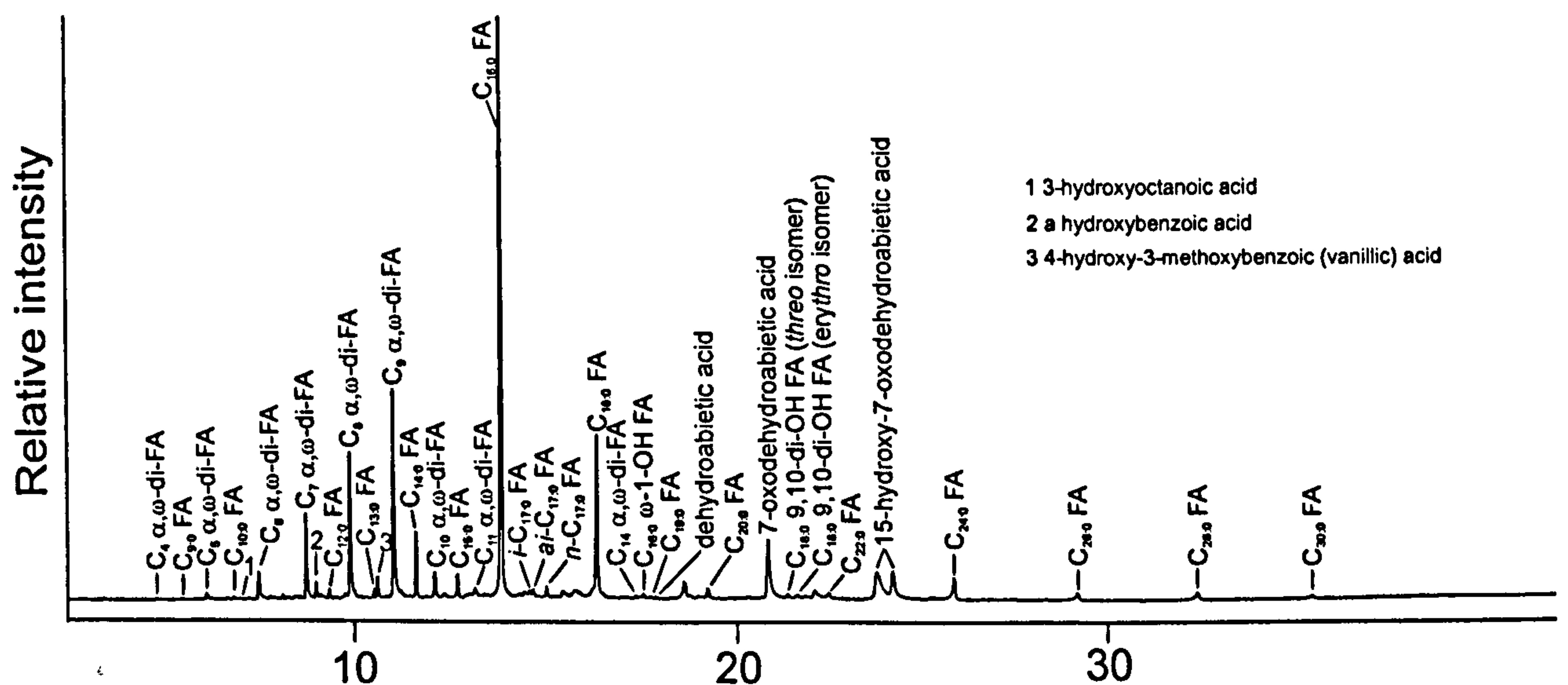


Figure 5.8b Total ion chromatogram from the GC/MS analysis of the acid fraction of 'resin' from the thoracic cavity [1] of the Third Intermediate Period female adult, XXIst-XXVth dynasty (c. 1069-664 B.C.).

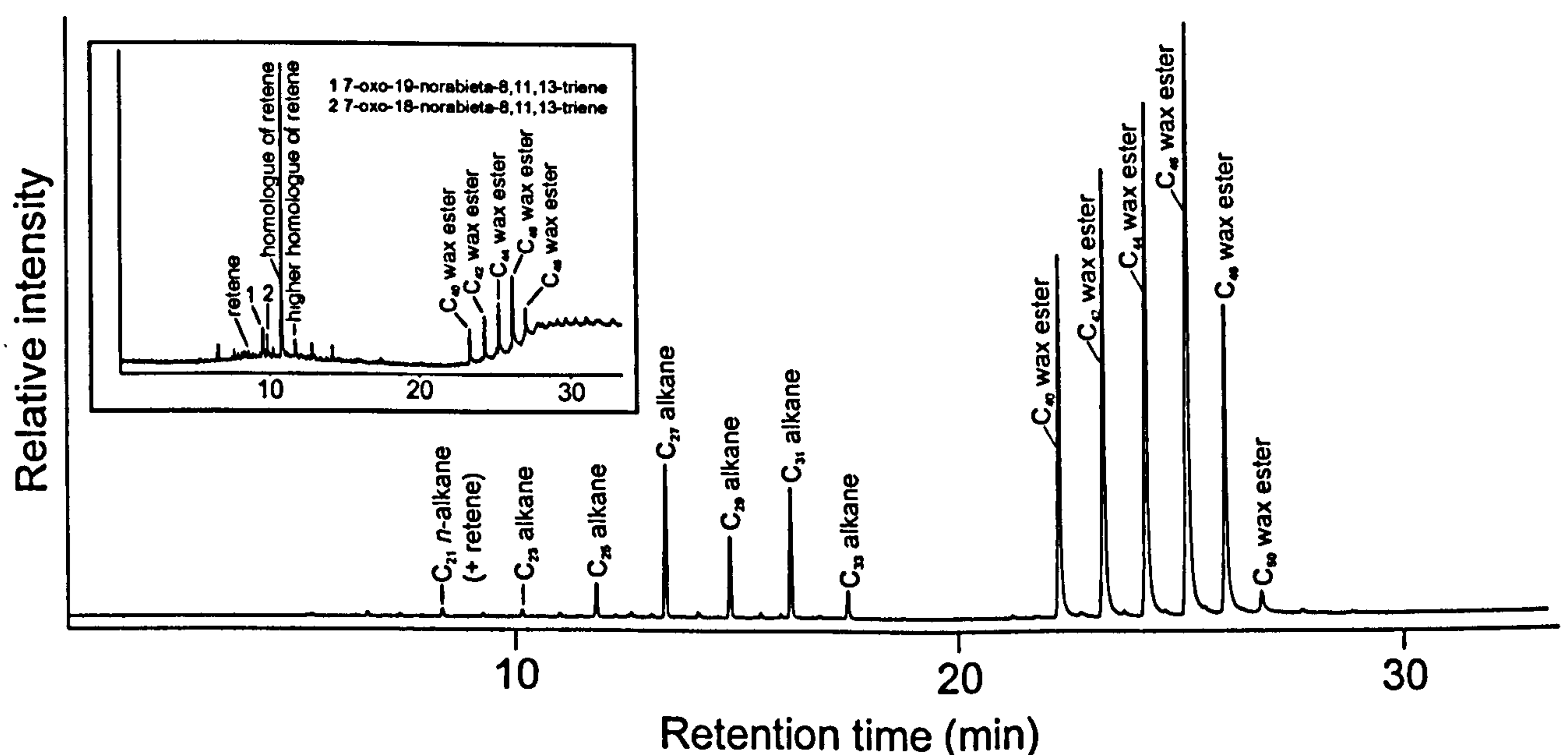


Figure 5.8c Total ion chromatograms from the GC/MS analyses of the neutral-hexane fraction, and (inset) neutral-DCM fraction of 'resin' from the thoracic cavity [1] of the Third Intermediate Period female adult, XXIst-XXVth dynasty (c. 1069-664 B.C.).

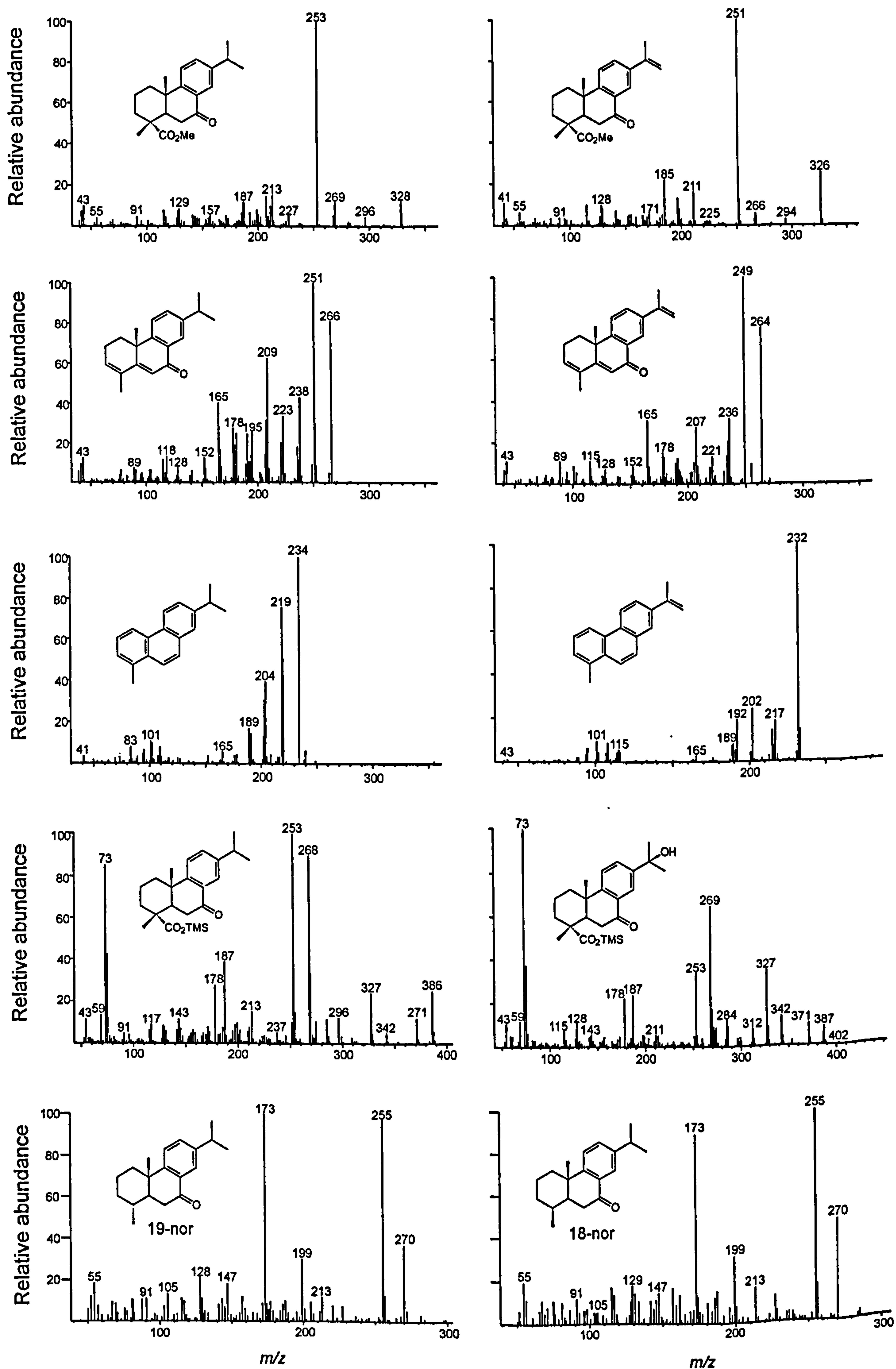


Figure 5.9 Mass spectra of the diterpenoid compounds identified in the TD-GC/MS and solvent extracts of 'resin' from the thoracic cavity [1] of the Third Intermediate Period female adult, XXIst-XXVth dynasty (c. 1069-664 B.C.).

unusual C_{8:0} 3-hydroxy- and C_{16:0} 15-hydroxy- fatty acids (as their TMS ether, TMS ester (see discussion). The aromatic acids 4?-hydroxy benzoic and vanillic were observed as minor constituents. Diterpenoids (as their TMS derivatives) were evidenced by a number of abietane type acids (see Fig. 5.9), and although dehydroabietic acid was only a trace component, the oxidised acids 7-oxodehydroabietic acid (m/z 73, 187, 253 (base peak), 268 and M^+ 386) and 15-hydroxy-7-oxo-dehydroabietic acid (as both its free hydroxy, TMS ester (m/z 73 (base peak), 269, 284 and $[M-15]^+$ 387) and its fully trimethylsilylated derivative (m/z 73, 285, 341 and $[M-15]^+$ 459 (base peak)) were significant components of the acid fraction, each of similar abundance to the C_{14:0} fatty acid.

GC/MS – Neutral fraction

a) Hexane fraction:

The results for the neutral-hexane fraction analysed by GC/MS are shown in Figure 5.8c. A series of alkanes (C₂₁ to C₃₃ maximising at C₂₇), with odd-over-even preference and wax esters (C₄₀ to C₅₀, maximising at C₄₆) dominated the chromatogram. The wax esters contained the C_{16:0} acyl group with small quantities of the C_{18:0} group (see discussion). Retene was also present as a minor component, coeluting with the C₂₁ alkane. The hexane fraction constituted a significant proportion of the total solvent extractable material (29%).

b) DCM fraction:

The results of the neutral-DCM fraction analysed by GC/MS are shown in Figure 5.8c, inset. The chromatogram was dominated by diterpenoids, including retene, two higher homologues of retene (one being the major component of the fraction), and the two epimers 7-oxo-19-norabieta-8,11,13-triene and 7-oxo-18-norabieta-8,11,13-triene (see Fig. 5.9). As would be expected these diterpenoids were also observed in the TD-GC/MS analysis. The DCM fraction constituted very little of the total solvent extractable material (<1%).

5.4.7 Female Adult 'Neskhons', XXIInd dynasty (c.945-715 BC), Third Intermediate Period, Thebes (H.5062)

5.4.7.1 'Resin'-soaked outer wrapping from left side of neck/cervical vertebrae [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.10a. A series of monocarboxylic acids (C_8 to C_{18}) was detected, the major components being $C_{16:0}$, $C_{18:0}$ and $C_{14:0}$ in decreasing order of abundance. A $C_{18:1}$ and two $C_{18:2}$ fatty acids were also identified, albeit as minor components, and there were unusually high relative abundances of the short chain fatty acids ($C_{8:0}$ to $C_{12:0}$). Also notable were $C_{16:0}$ γ - and δ -lactones characterised by m/z 85, 192 and $[M-H_2O]^+$ 236; and m/z 99, 192 and $[M-H_2O]^+$ 236 respectively (see Fig. 5.11), and the C_{18} γ - and δ -lactones characterised by m/z 85, 220 and $[M-H_2O]^+$ 264; and m/z 99, 220 and $[M-H_2O]^+$ respectively (see Fig. 5.11). Steroidal compounds (see Fig. 5.11) of both an animal and plant origin were detected, cholesta-3,5,7-triene and cholesta-3,5-dien-7-one indicating an animal source, and stigma-3,5,7,22-tetraene and stigma-3,5,7-triene confirming a significant plant component in this sample. The presence of levoglucosan, and furan and pyran derivatives, suggests a possible sugar/plant gum origin, although the former may derive from hydrolysed cellulose present in the degraded linen wrappings. As shown in the two XVIIth dynasty mummies, intact cellulose does not produce levoglucosan and the furan/pyran derivatives at the thermal desorption temperatures used in this study, breakdown of the cellulose only taking place at the 610°C employed for pyrolysis. Minor components included the C_{27} , C_{29} and C_{31} n -alkanes and the two oxidised diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate. A number of sesquiterpenoids with a cadalene carbon skeleton were also detected, along with vanillic acid and at later retention times (73.5 to 95.0 mins) a complex pattern of triterpenoids was observed, the major components being 11(?),28-bis-norolean-17-en-3-one (M^+ 396 and base peak m/z 163) and, tentatively, 11-noroleanonic acid (M^+ 440 and base peak m/z 163) (see discussion). A number of these triterpenoids were ocotillone-type molecules (base peak m/z 143) and their dehydrated analogues (base peak m/z 125). Of those tentatively identified were 20,24-epoxy-25-hydroxy-28(?)-nordammaran-3-one and its dehydrated derivative 20,24-epoxy-28(?)-nordammar-25-en-3-one (two isomers), and 3-oxo-25,26,27,28-tetranordammarano-20,24-lactone (two isomers) (see Fig. 5.11). Two highly degraded triterpenoids were also tentatively identified as 22,23,24,25,26,27,28-heptanordammaran-3,20-dione (M^+ and base peak 344) and 20-hydroxy-25,26,27,28-tetranordammaran-3-one (M^+ and base peak 388). These last two

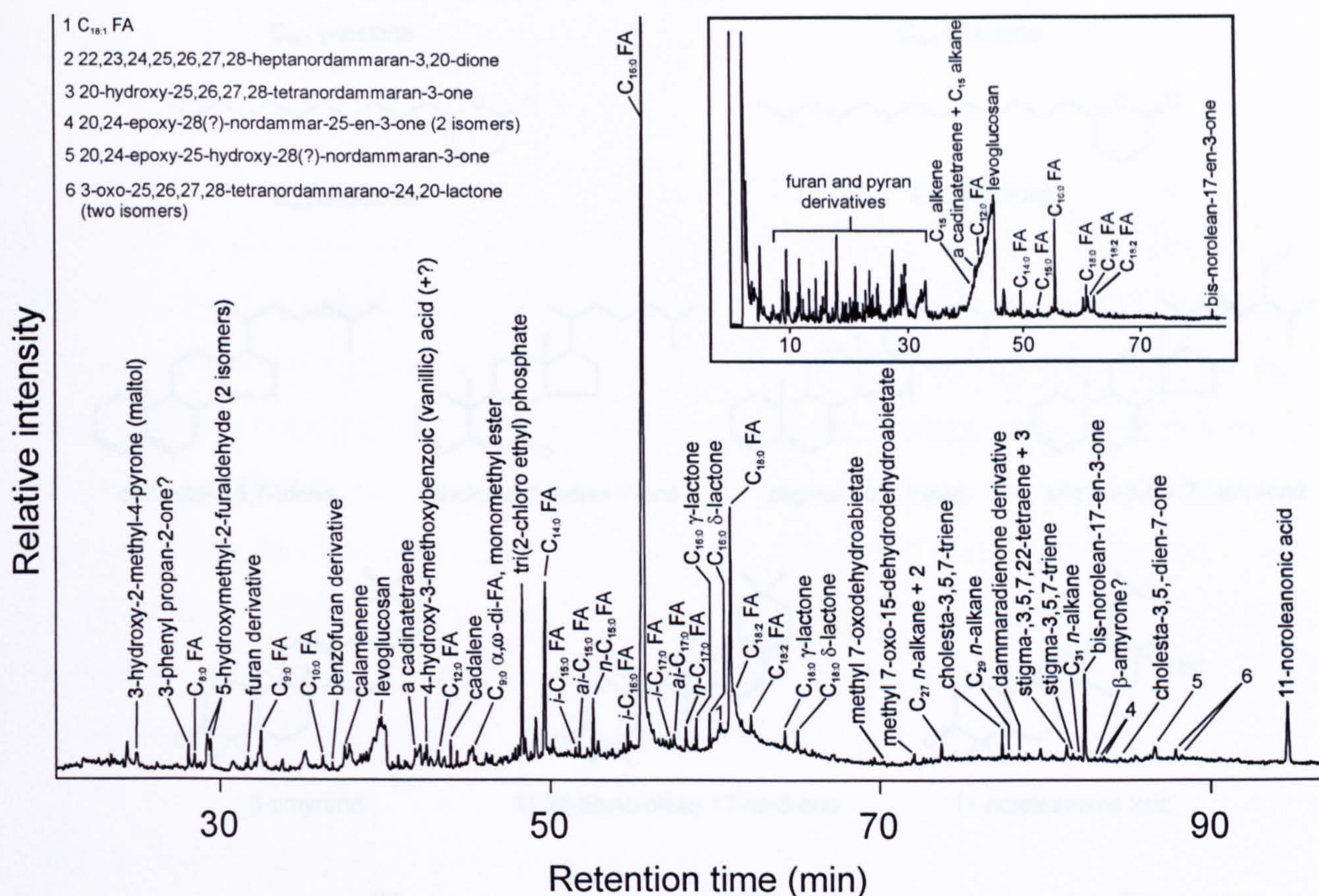


Figure 5.10a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin'-soaked outer wrapping from the left side of the neck/cervical vertebrae [1] of the Third Intermediate Period female adult 'Neskhnos', XXIInd dynasty (c. 945-715 B.C.).

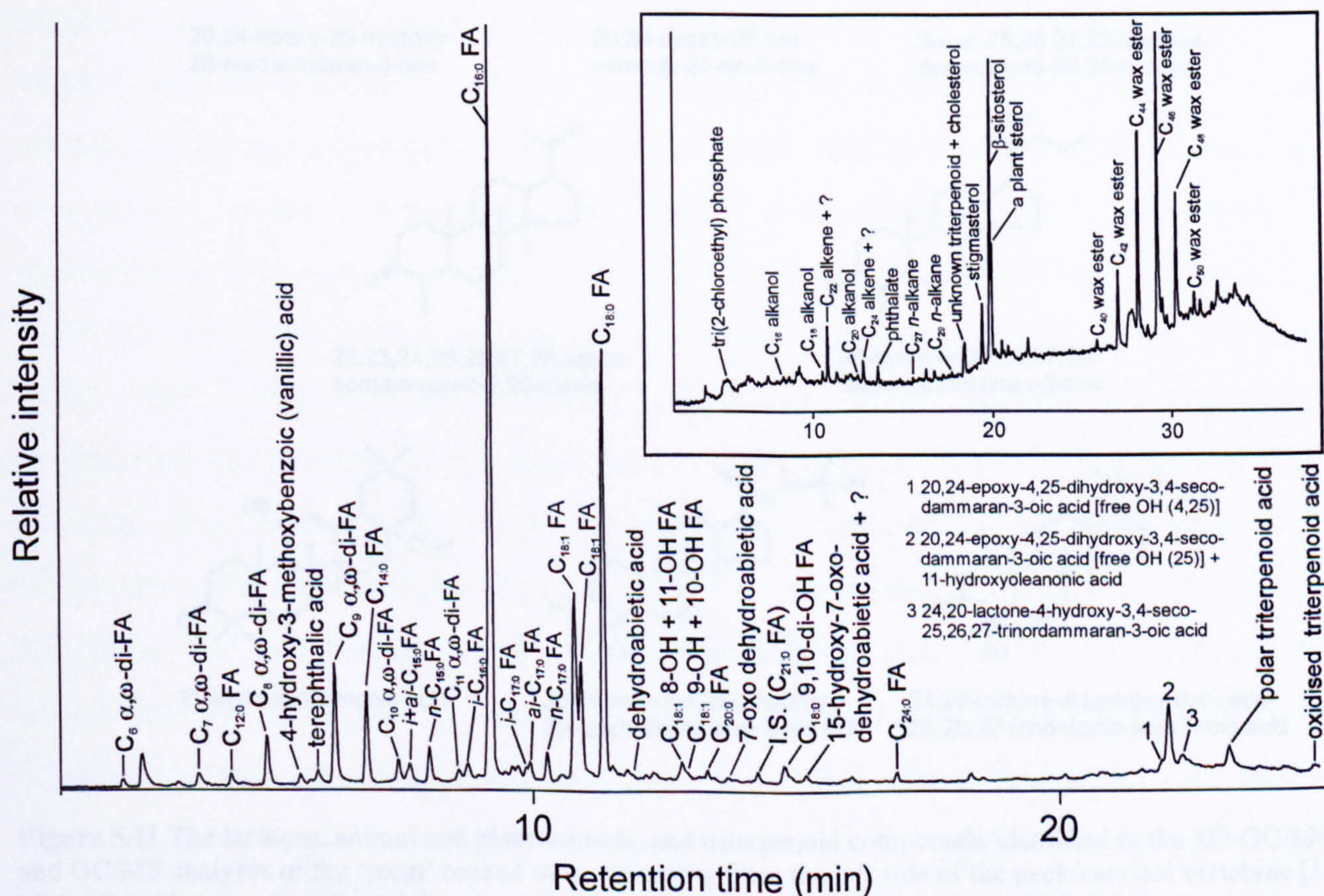


Figure 5.10b Total ion chromatograms from the GC/MS analyses of the acid fraction, and (inset) neutral fraction of 'resin'-soaked outer wrapping from the left side of the neck/cervical vertebrae [1] of the Third Intermediate Period female adult 'Neskhnos', XXIInd dynasty (c. 945-715 B.C.).

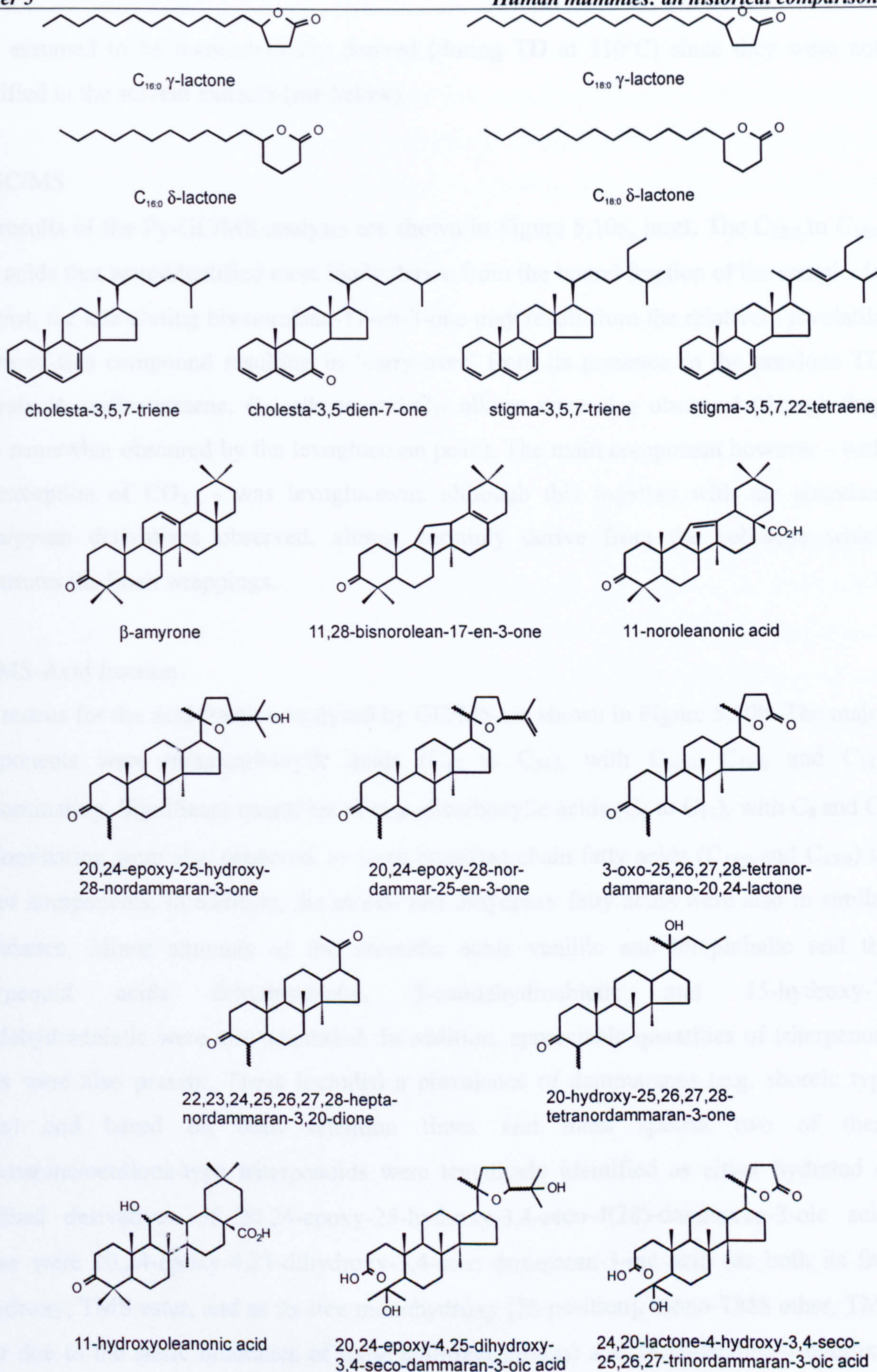


Figure 5.11 The lactones, animal and plant steroids, and triterpenoid compounds identified in the TD-GC/MS and GC/MS analyses of the 'resin' soaked outer wrapping from the left side of the neck/cervical vertebrae [1] of the Third Intermediate Period female adult 'Neskhn's', XXIInd dynasty (c. 945-715 B.C.).

were assumed to be thermolytically derived (during TD at 310°C) since they were not identified in the solvent extracts (see below).

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.10a, inset. The C_{12:0} to C_{18:0} fatty acids that were identified most likely derive from the bound fraction of the sample. In contrast, the late eluting bis-norolean-17-en-3-one may result from the relatively involatile nature of this compound resulting in 'carry-over' from its presence in the previous TD analysis. A cadinatetraene, C₁₅ alkene and C₁₅ alkane were also observed (though they were somewhat obscured by the levoglucosan peak). The main component however - with the exception of CO₂ - was levoglucosan, although this together with the abundant furan/pyran derivatives observed, almost certainly derive from the cellulose which constitutes the linen wrappings.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.10b. The major components were monocarboxylic acids (C₁₂ to C₂₄), with C_{16:0}, C_{18:0} and C_{18:1} predominating. Significant quantities of α,ω -dicarboxylic acids (C₆ to C₁₁), with C₈ and C₉ predominating were also observed, as were branched chain fatty acids (C_{15:0} and C_{17:0}) as minor components. In addition, the mono- and dihydroxy fatty acids were also in similar abundance. Minor amounts of the aromatic acids vanillic and terephthalic and the diterpenoid acids dehydroabietic, 7-oxodehydroabietic and 15-hydroxy-7-oxodehydroabietic were also identified. In addition, appreciable quantities of triterpenoid acids were also present. These included a prevalence of dammaranes (e.g. shoreic type acids) and based on both retention times and mass spectra two of these dammarane/octillone-type triterpenoids were tentatively identified as either hydrated or oxidised derivatives of 20,24-epoxy-25-hydroxy-3,4-seco-4(28)-dammaran-3-oic acid. These were 20,24-epoxy-4,25-dihydroxy-3,4-seco-dammaran-3-oic acid (as both its free dihydroxy, TMS ester, and as its free monohydroxy [25 position], mono-TMS ether, TMS ester due to the steric hindrance of these hydroxyl groups) and 24,20-lactone-4-hydroxy-3,4-seco-25,26,27-trinordammaran-3-oic acid (as its TMS ether, TMS ester) (see Figure 5.11). The two derivatives of the former compound were characterised by *m/z* 73, 125 and 143 (base peak), and the latter by *m/z* 73, 99 and 315. The presence of these compounds is tentatively supported by the TD analysis where the nor-compounds of the previously observed triterpenoids 20,24-epoxy-25-hydroxy-dammaran-3-one and 3-oxo-25,26,27-

trisnordammarano-20,24-lactone (van der Doelen et al. 1998, p.249-264) were detected, apparently being formed in the probe at 310°C via the cyclisation (loss of HCHO and H₂O) of the corresponding acids. The relative abundances of these compounds would seem to agree with this, but further work is necessary in order to fully confirm this conclusion. Again tentatively identified were a number of highly oxidised oleanane/ursane triterpenoids, the major compound identified as 11-hydroxyoleanonic acid (as its TMS ether, TMS ester) and characterised by m/z 73, 161, 189, 203, 279 and 309. Unfortunately the abundances of the triterpenoid acids were too low to obtain good mass spectra which could aid their positive identification.

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.10b, inset. The chromatogram was dominated by the plant sterols, β -sitosterol (as its TMS ether, characterised by m/z 129 (base peak), 357, 381, 396 and M^+ 486) and stigmasterol (as its TMS ether, characterised by m/z 213, 255, 351, 394 and M^+ 484), and a series of wax esters (C₄₀ to C₅₀) maximising at C₄₆. The wax esters were dominated by the C_{16:0} acyl group although significant amounts of the C_{18:0} acyl group were also present. C₂₇ and C₂₉ *n*-alkanes (also observed in the TD analysis) were identified, although their presence as only very minor components, along with the observation of the more dominant C₂₂ and C₂₄ alkenes suggests that hydrocarbons potentially diagnostic of a particular wax were highly degraded in this sample leaving the precise origin and nature of the wax uncertain. Minor components present in this fraction included alkanols (C₁₆ to C₂₀) and an unidentified triterpenoid coeluting with cholesterol (as its TMS ether). The neutral fraction constituted an appreciable proportion of the total extract (18%).

5.4.7.2 'Resin'-soaked wrapping from left hip/innominate bone [2]

TD-GC/MS

The results of the TD-GC/MS analysis displayed a series of monocarboxylic acids (C₁₄ to C₁₈) were detected, the major components being C_{16:0}, C_{18:0} and C_{14:0} in order of decreasing abundance. Two C_{18:2} fatty acids were also identified, as were the C_{16:0} and C_{18:0} γ - and δ -lactones as minor components. These fatty acid compounds were very similar in both quantity and quality to those of the previous sample from Neskons (5.4.7.1). The cholesterol derivative cholesta-3,5,7-triene was observed, although the derivatives of the plant sterols were not detected. Although less abundant than in 5.4.7.1, levoglucosan, and furan and pyran derivatives were identified, again suggesting a possible sugar/plant gum

origin. A cadinatetraene and cadalene were observed along with the later eluting triterpenoid bis-norolean-17-en-3-one. Present as trace components were methyl 7-oxodehydroabietate, methyl 7-oxo-15-dehydrodehydroabietate and the C₂₇ *n*-alkane. The TD profiles of the two wrapping samples suggest that the same embalming agents were applied to both the neck and hip.

Py-GC/MS

Py-GC/MS analysis identified the C_{14:0}, C_{16:0} and C_{18:0} fatty acids, most likely deriving from the bound fraction of the sample. A cadinatetraene, C₁₅ alkene and C₁₅ alkane were also observed (though they were somewhat obscured by the levoglucosan peak) and as in the wrappings from the neck of Neskons (5.4.7.1) levoglucosan was a major component, along with significant amounts of furan/pyran derivatives, and deriving almost certainly from the cellulose-based wrappings.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS revealed a series of monocarboxylic acids (C₁₂ to C₂₄), with C_{16:0}, C_{18:0} and C_{14:0} predominating, and C_{18:1} present only as a minor component. The distribution of these acids was similar to that obtained using TD. High abundances of α,ω -dicarboxylic acids (C₆ to C₁₃), with C₈ and C₉ predominating were also observed, indicating the highly oxidised nature of the sample. However, the mono- and dihydroxy carboxylic acids, although present, were relatively minor components, as were the C_{15:0} and C_{17:0} branched chain fatty acids. The aromatic acids hydroxybenzoic, α -hydroxyhydrocinnamic, vanillic and terephthalic were also identified as minor components, with trace quantities of dehydroabietic, 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid. The same dammarane and oleanane/ursane type triterpenoid acids (as their TMS derivatives) were also observed, albeit at lower abundance than those from the neck wrappings.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS revealed a series of *n*-alkanes (C₂₅ to C₃₃ maximising at C₃₃) with an odd-over-even preference and wax esters (C₄₂ to C₅₀ maximising at C₄₆). The wax esters are dominated by the C_{16:0} acyl group although significant amounts of C_{18:0} were also present, particularly in the less abundant components (C₄₂ and C₅₀). The only other components of any significance were the C_{16:0} and C_{18:0} 1-monoacylglycerols. The neutral components were relatively minor (5%) compared to the

major constituents of the acid fraction, therefore the absence of the alkanes (except C₂₇ as a trace component) from the TD-GC/MS (at least in any significant abundance) is not unexpected.

5.4.8 Male Adult 'Pedeamun', XXVIth-XXVIIth dynasty (c.664-404 BC), Late Period, Thebes (1953.72)

5.4.8.1 'Resin' from inside cartonnage at back of cranium [1].

TD-GC/MS

The results of the TD-GC/MS analysis displayed a complex profile with many components present. Of those identified were C_{14:0}, C_{16:0} and C_{18:0} fatty acids, the second of which was the major component detected by TD. A series of *n*-alkanes (C₂₅ to C₃₁, maximising at C₂₇) with an odd-over-even preference were also present in abundance, with moderate amounts of the γ - and δ -lactones and trace amounts of methyl 7-oxodehydroabietate and. methyl 7-oxo-15-dehydrodehydroabietate.

Py-GC/MS

The results of the Py-GC/MS analysis revealed a series of alkene/alkane doublets (C₉ to C₂₃) with longer chain alkenes also significant (C₂₄ to C₃₀ with an even-over-odd preference) (for the likely origin of these see discussion below) and the C_{16:0} fatty acid.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS displayed a series of monocarboxylic acids (C₁₄ to C₃₀), with C_{16:0} and C_{18:0} predominating. The long chain saturated fatty acids were significant constituents of the acid fraction, with lignoceric acid predominating. Of the more polar carboxylic acids the α,ω -dicarboxylic acids (C₅ to C₁₁) were particularly abundant with azelaic acid being the main component after palmitic acid. Relatively minor quantities of the C_{18:1} monohydroxy- and C_{18:0} 9,10-dihydroxy- carboxylic acids were also present. Vanillic acid was detected in moderate abundance with minor quantities of dehydroabietic and 7-oxodehydroabietic acids.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS revealed a series of *n*-alkanes (C₂₅ to C₃₃) and wax esters (C₄₀ to C₅₀ maximising at C₄₆), the latter being the major components in the chromatogram and consisting predominantly of the C_{16:0} acyl group.

Notably monohydroxy wax esters (C_{44} to C_{50}) were present as minor components, characterised by m/z 117, and eluting just after the wax esters of the corresponding carbon number. The C_{28} and C_{30} *n*-alkanols were also minor constituents. Significant amounts of $C_{16:0}$ and $C_{18:0}$ fatty acid methyl esters were also identified, although it is not yet certain whether they derive from the original sample or are a result of *in situ* methylation during the extraction stage, due to the sample's highly acidic nature (i.e. matrix effects). It should also be noted that the extraction procedure does not normally produce such artefacts. The neutral fraction constituted almost half (45%) of the total solvent extractable material.

5.4.8.2 'Resin' from top of cranium [3].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.12a. Of those peaks identified were a series of monocarboxylic acids (C_{14} to C_{18}) with $C_{16:0}$ predominating and moderate amounts of the $C_{16:0}$ γ - and δ -lactones. The C_{25} to C_{31} *n*-alkanes, maximising at C_{27} with an odd-over-even preference, were once again present.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.12a, inset. The pyrogram revealed a series of alkene/alkane doublets (C_9 to C_{23}) with lesser amounts of longer chain alkenes (C_{24} to C_{30} with an even-over-odd preference). These, along with the $C_{16:0}$ fatty acid were the major components (excluding CO_2) of the bound/polymeric material.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.12b, inset. A range of carboxylic acids were identified, the chromatogram being dominated by a series of monocarboxylic acids (C_{14} to C_{30}). $C_{16:0}$ and $C_{18:0}$ were the main two components of the acid fraction in decreasing order of abundance, unsaturated acids being absent. Saturated long-chain acids in the $C_{22:0}$ and $C_{30:0}$ carbon number range with lignoceric predominant were present. The profile of the monocarboxylic acids was comparable with the TD profile obtained for this sample. A series of α,ω -dicarboxylic acids in the C_6 to C_{11} carbon number range were present in significant abundance, together with lesser amounts of mono- and dihydroxy carboxylic acids. Hydroxybenzoic, terephthalic and vanillic acids were observed (the latter in moderate abundance), as were dehydroabietic and 7-oxo-dehydroabietic acids, albeit only as trace components.

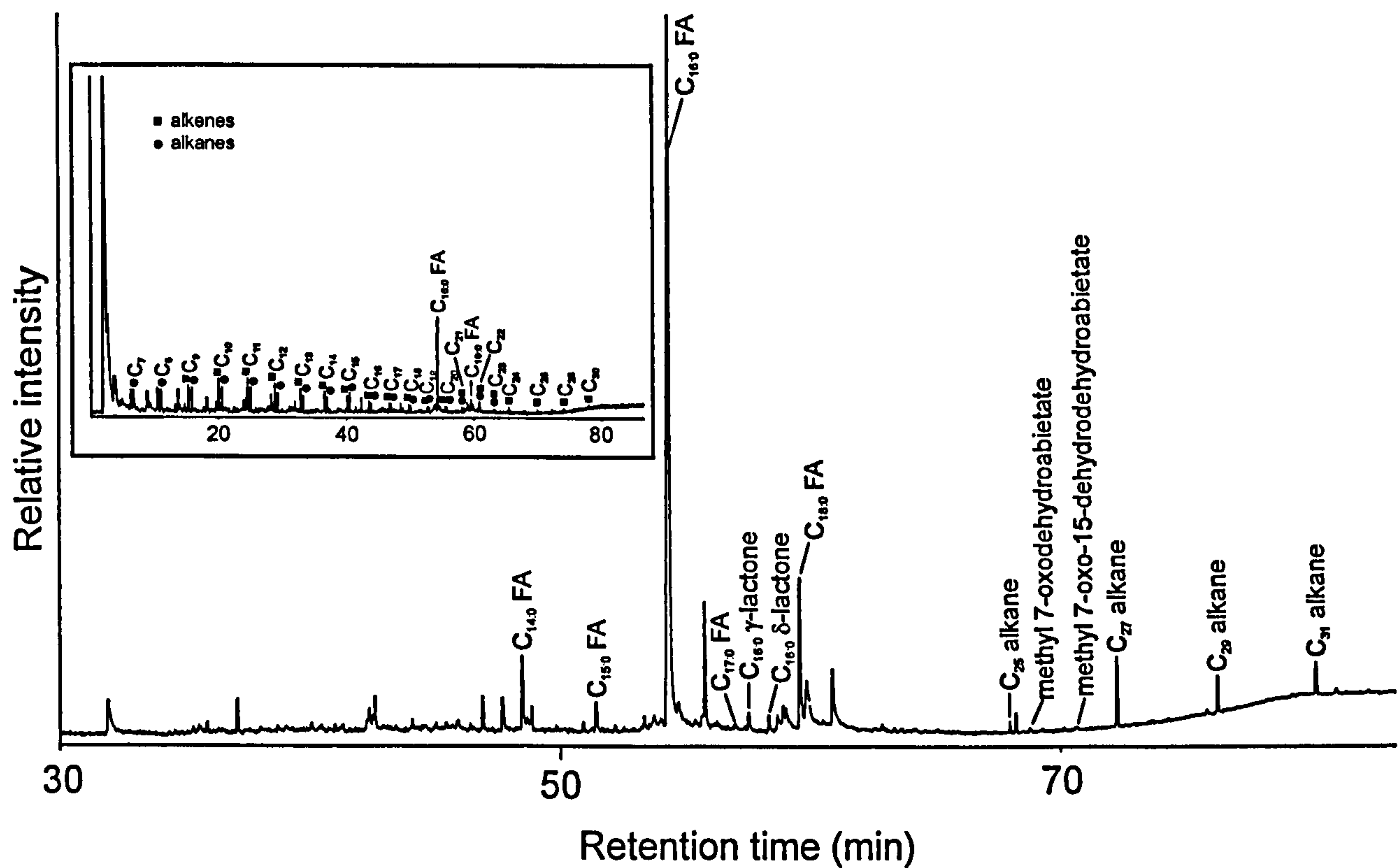
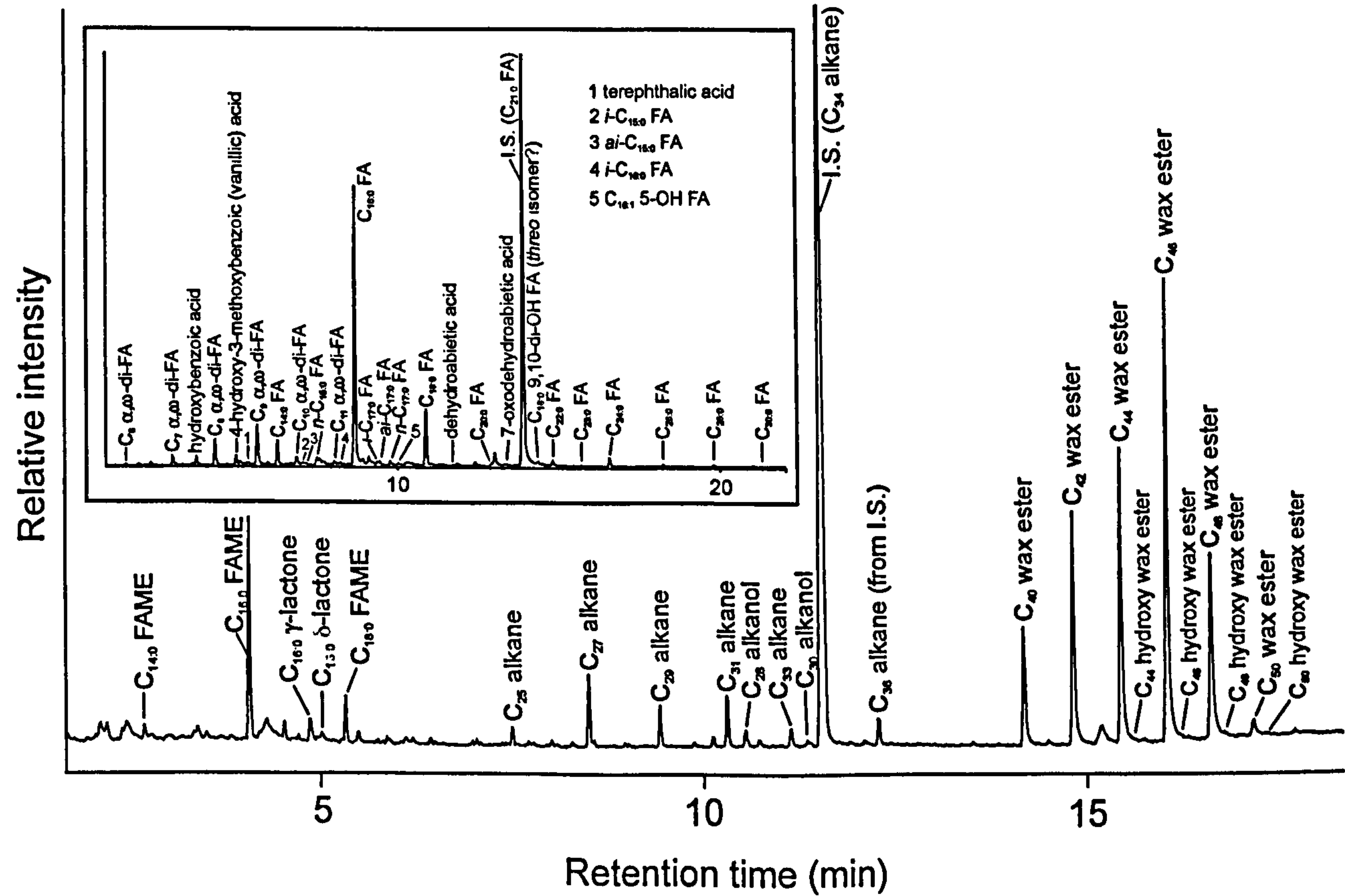


Figure 5.12a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin' from the top of the cranium [3] of the Late Period male adult 'Pedeamun', XXVIth-XXVIIth dynasty (c. 664-404 B.C.).



GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.12b. A series of *n*-alkanes (C_{25} to C_{33} maximising at C_{27}) with an odd-over-even preference were observed, along with wax esters in the C_{40} to C_{50} carbon number range, C_{46} predominating. The C_{40} , C_{44} , C_{46} and C_{48} wax esters contained predominantly the $C_{16:0}$ acyl group (m/z 257), with minor amounts of $C_{18:0}$ (m/z 285), the C_{42} and C_{50} wax esters also consisting predominantly of the $C_{16:0}$ acyl group, but with a more significant proportion of $C_{18:0}$. Monohydroxy wax esters (C_{44} to C_{50}) were also detected as minor components, characterised by m/z 117 and eluting just after the wax esters of corresponding carbon numbers. Significant amounts of $C_{16:0}$, $C_{18:0}$ and $C_{14:0}$ fatty acid methyl esters were also identified, although it is not certain at this stage whether they derive from the original sample or are the result of *in situ* methylation during the extraction stage, due to the sample's highly acidic nature (i.e. matrix effect). However, it should be noted that the extraction procedure does not normally produce such artefacts. In addition to the aforementioned constituents were the $C_{16:0}$ γ - and δ -lactones, not normally observed in the solvent extracts, and also observed in the TD-GC/MS. The neutral fraction constituted almost half (49%) of the total solvent extractable material.

5.4.8.3 'Resin' from upper edge of cartonnage opposite right cheek/zygomatic bone [5].

[nb. this minute sample was taken to demonstrate the potential of sequential TD-GC/MS and Py-GC/MS, the sample size of particular interest to the curator and conservator of the museum in question, although no solvent extraction procedures could be carried out on such a small amount of material].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.13a. The profile was very similar to the TD-GC/MS analyses from the two other samples (5.4.8.1 & 5.4.8.2). Of the components identified were the monocarboxylic acids $C_{16:0}$, $C_{18:0}$ and $C_{14:0}$ in decreasing order of abundance. Also present as major constituents were a series of *n*-alkanes (C_{25} to C_{31} , with C_{27} predominating) with an odd-over-even preference and their distribution very similar to 5.4.8.2 (see Figure 5.12). Moderate amounts of the $C_{16:0}$ γ - and δ -lactones and the two diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate were also observed.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.13b. The pyrogram revealed a series of alkene/alkane doublets (C_9 to C_{23}), with longer chain alkenes also dominant (C_{24}

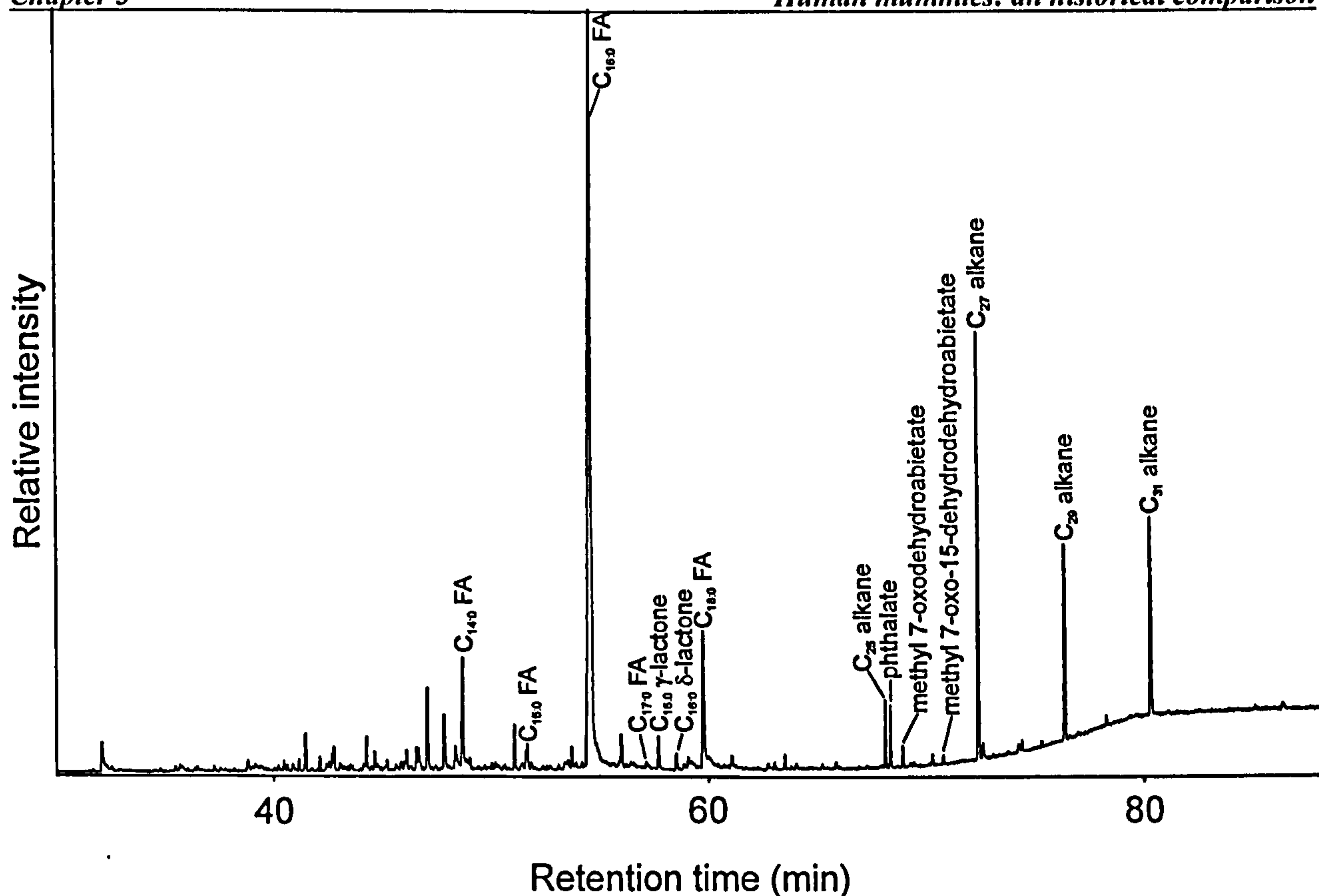


Figure 5.13a Total ion chromatogram of the thermal desorption profile (310°C/10s) of 'resin' from the upper edge of the cartonnage opposite the right cheek/zygomatic bone [5] of the Late Period male adult 'Pedeamun', XXVIth-XXVIIth dynasty (c. 664-404 B.C.).

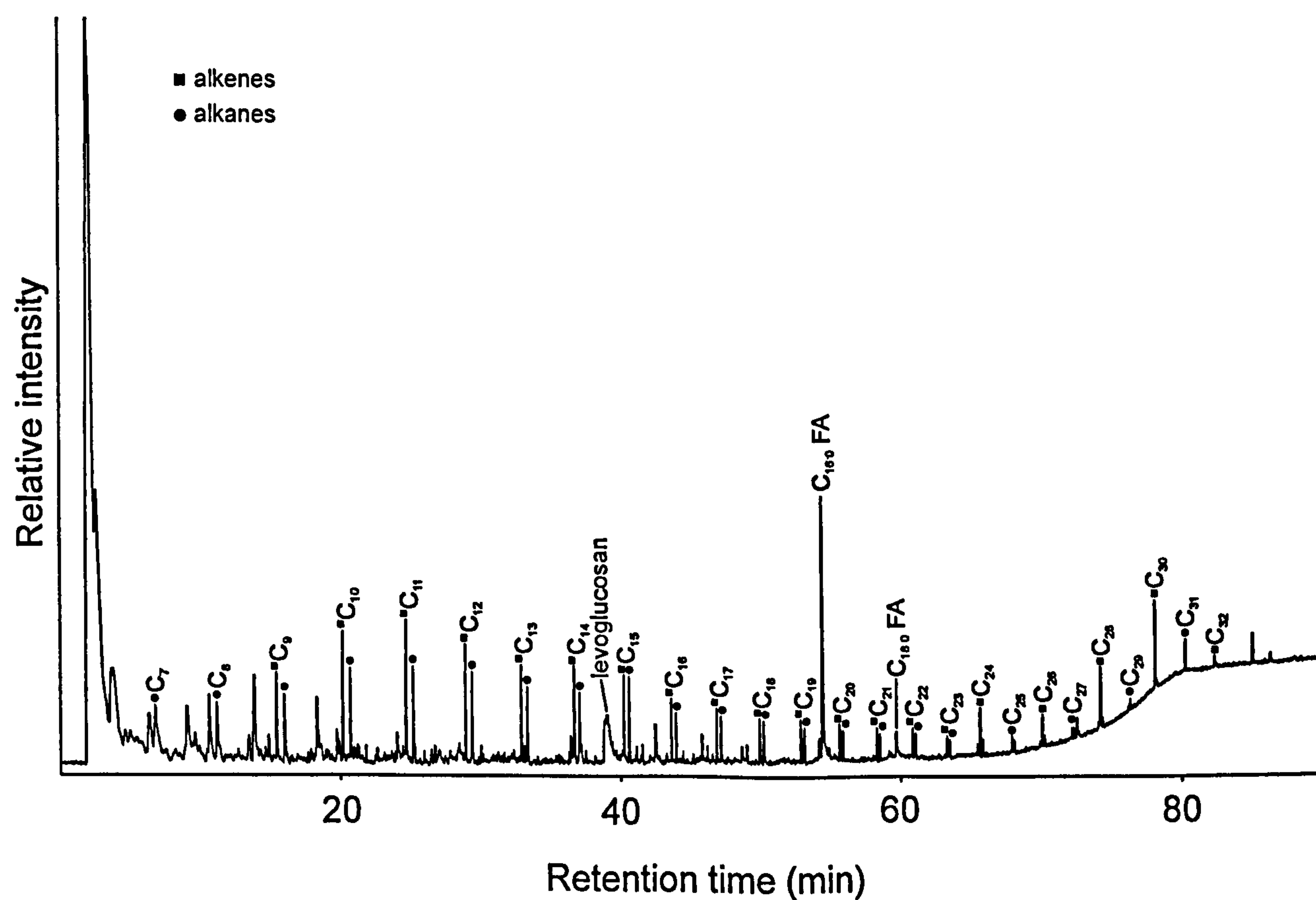


Figure 5.13b Total ion chromatogram of the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin' from the upper edge of the cartonnage opposite the right cheek/zygomatic bone [5] of the Late Period male adult 'Pedeamun', XXVIth-XXVIIth dynasty (c. 664-404 B.C.).

to C₃₂, with an even-over-odd preference) (for the likely origin of these, see discussion), and the C_{16:0} fatty acid as the major components (excluding CO₂) of the bound/polymeric material.

5.4.9 Female Adult, Ptolemaic (c.332-30 BC), Thebes (1956.352)

5.4.9.1 'Resin' attached to linen thread from right ankle/talus [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.14a. The TD profile was dominated by a series of alkanes (C₂₃ to C₃₃ with C₂₇ predominating) and the C_{16:0} fatty acid. Also detected as a relatively minor component was the triterpenoid 28-norolean-17-en-3-one.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.14a, inset. The pyrogram was dominated by a series of alkene/alkane doublets (C₉ to C₂₃), with longer chain alkenes also dominant (C₂₄ to C₃₀) (for the likely origin of these, see discussion). The fatty acid C_{16:0} is also significant, being the major component (excluding CO₂) of the pyrogram. The triterpenoid 28-norolean-17-en-3-one was also detected in appreciable abundance.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.14b, inset. A number of aromatic acids were identified in moderate abundance, these were 4(?) -hydroxybenzoic, vanillic, terephthalic and 3,4?-dihydroxybenzoic (protocatechuic) acids (as their TMS derivatives). Of similar abundance were the C_{16:0} to C_{18:0} fatty acids. However, the main components of this fraction were triterpenoid acids of the oleanane and euphane type identified as their TMS derivatives. Moronic acid, characterised by m/z 73, 189 (base peak), 203, 307, 409 and M⁺ 526) was identified as the major acid component, along with lesser amounts of oleanonic (characterised by m/z 73, 189, 203 (base peak), 307, 408 and M⁺ 526) (see Figure 5.15). Also identified as their TMS esters were isomasticadienonic (m/z 73, 255 (base peak), 373, 408 and M⁺ 526) and masticadienonic (m/z 73, 257 (base peak), 375, 408 and M⁺ 526) acids based upon both their retention times and mass spectra. In addition, other triterpenoids containing additional functional groups were tentatively identified, based upon the retention times and mass spectra. These included 3-oxoolean-13-en-28-oic acid (two isomers) (m/z 73, 189 (base peak) and 203),

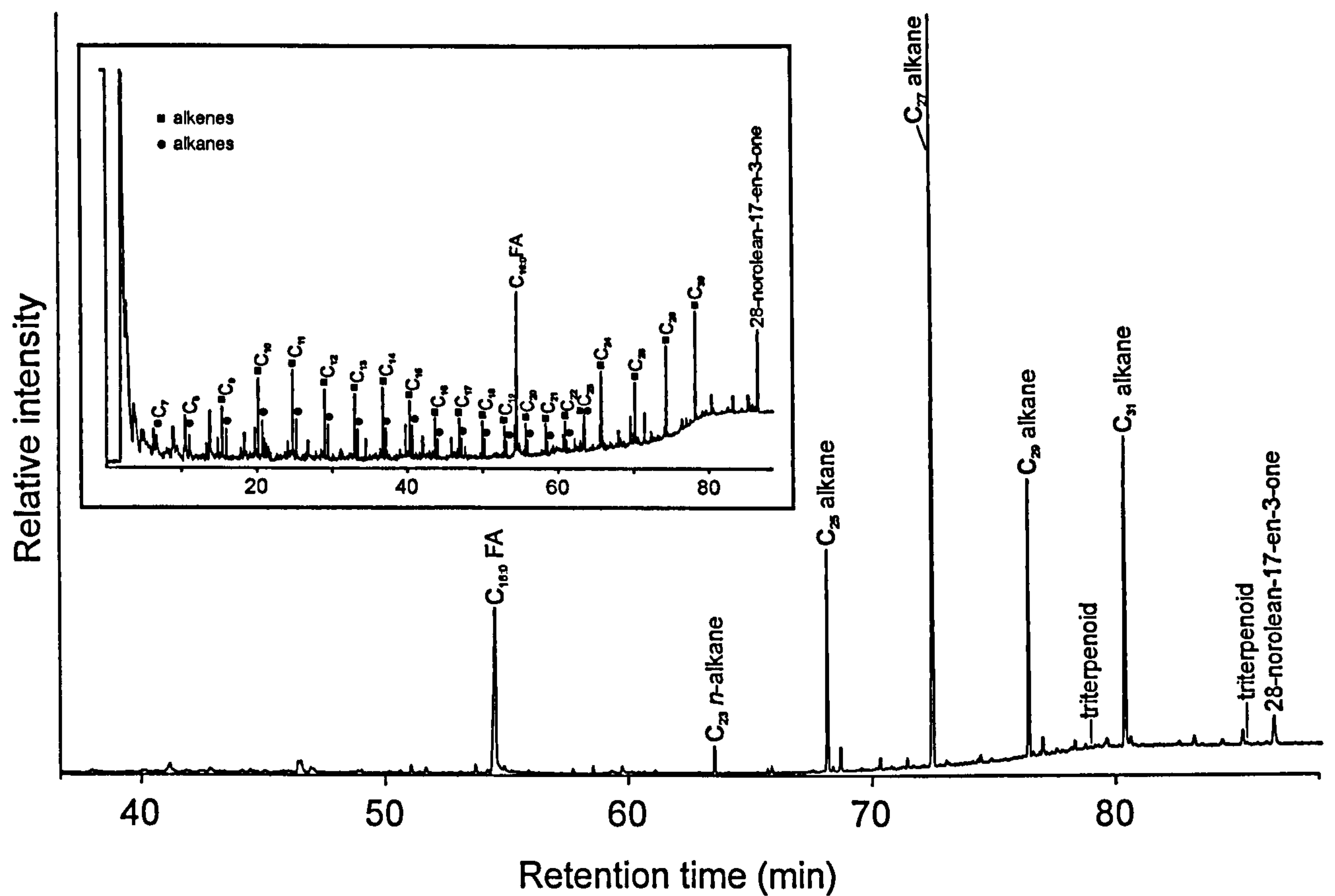


Figure 5.14a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of ‘resin’ attached to linen thread from the right ankle/talus [1] of the Ptolemaic female adult (c. 332-30 B.C.).

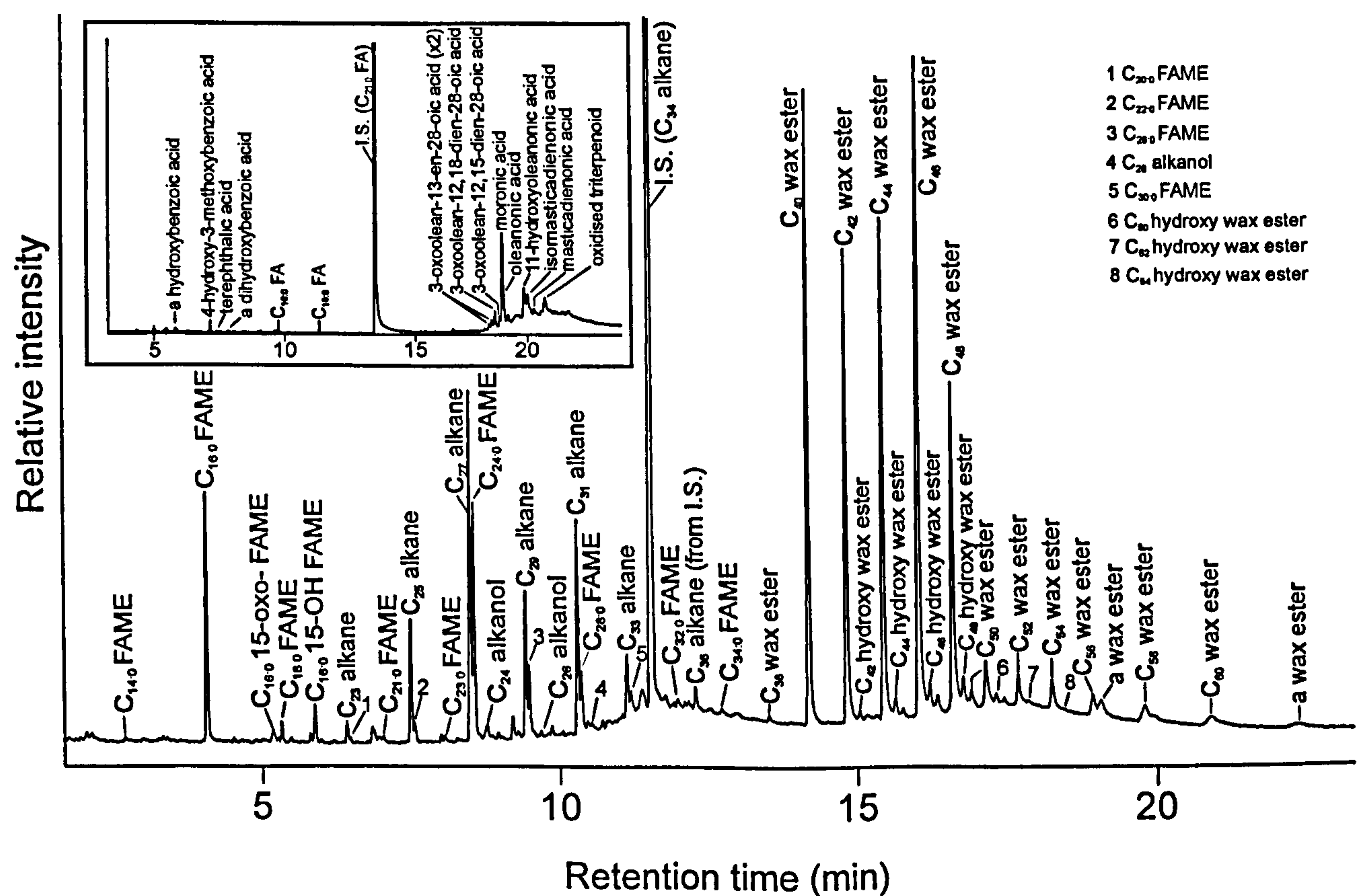
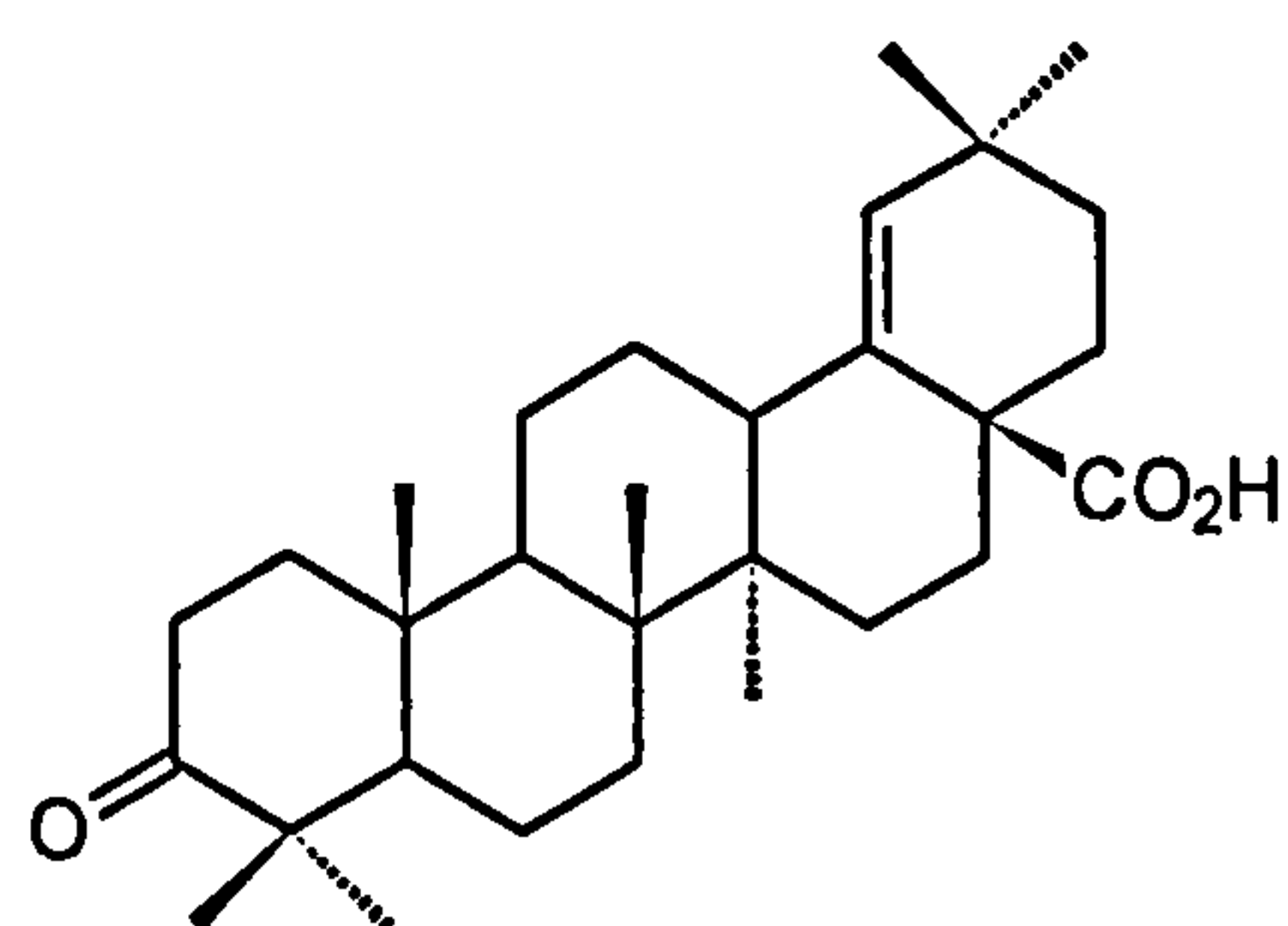
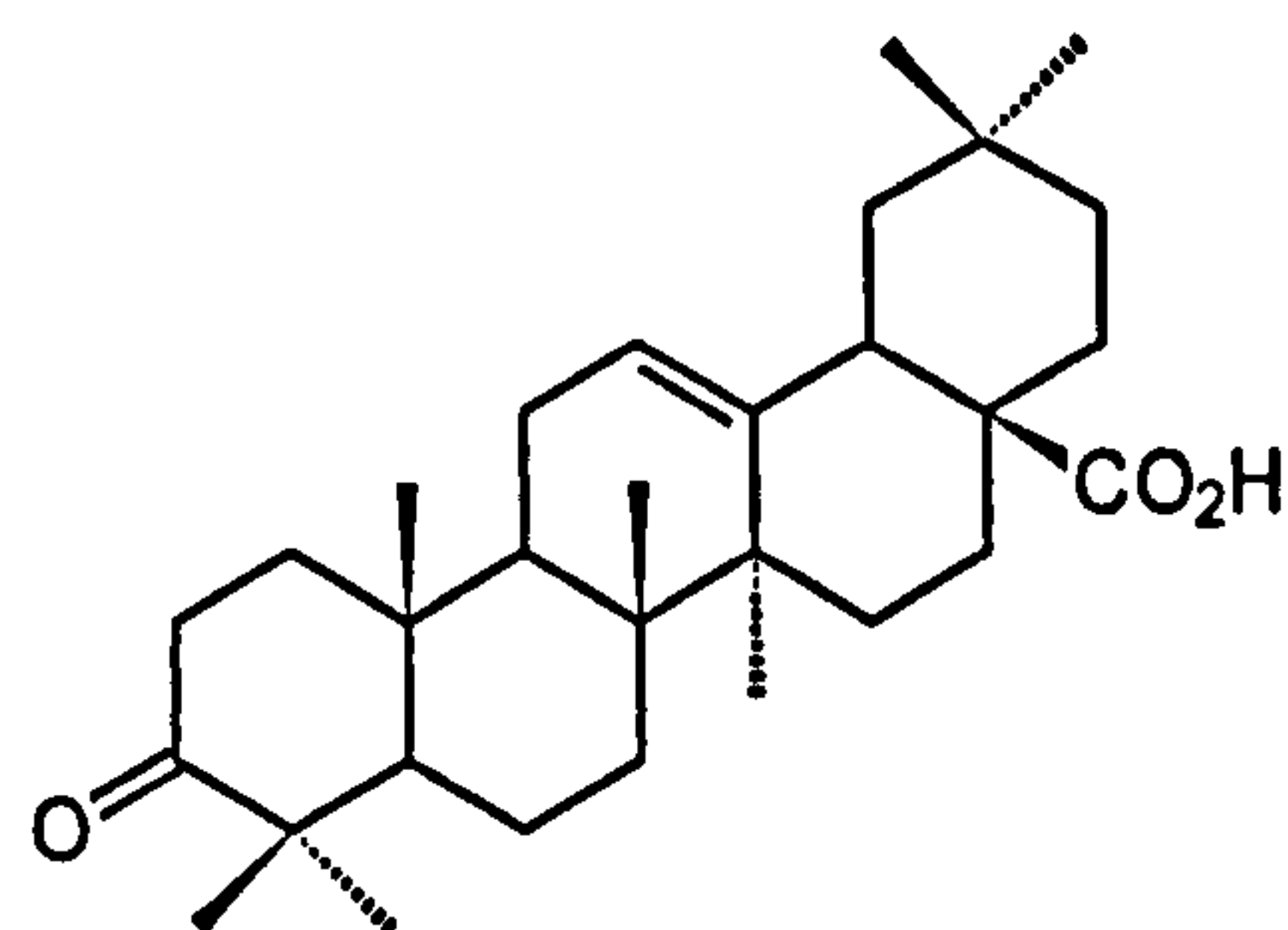


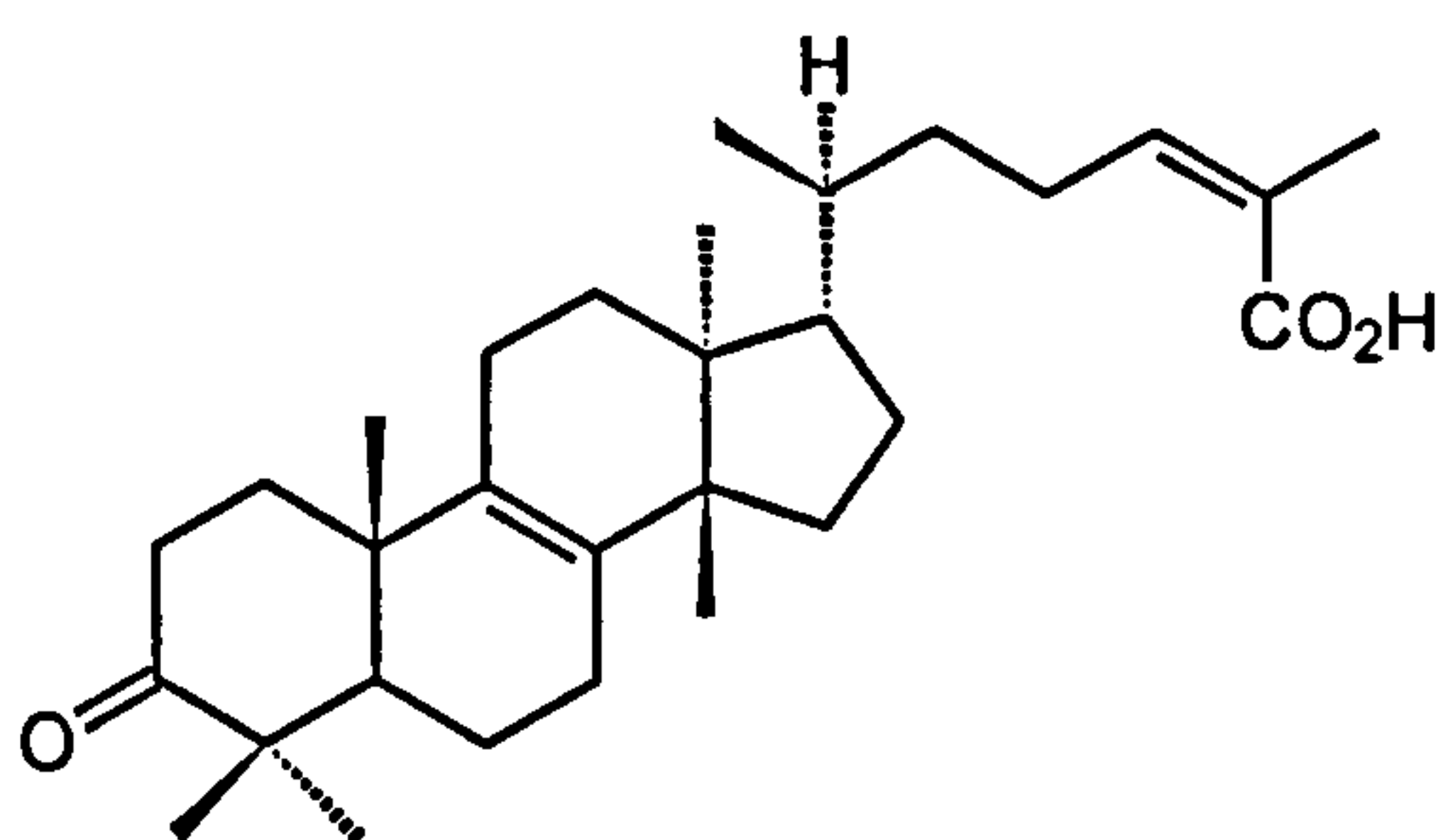
Figure 5.14b Total ion chromatograms from the GC/MS analyses of the neutral fraction, and (inset) acid fraction of ‘resin’ attached to linen thread from the right ankle/talus [1] of the Ptolemaic female adult (c. 332-30 B.C.).



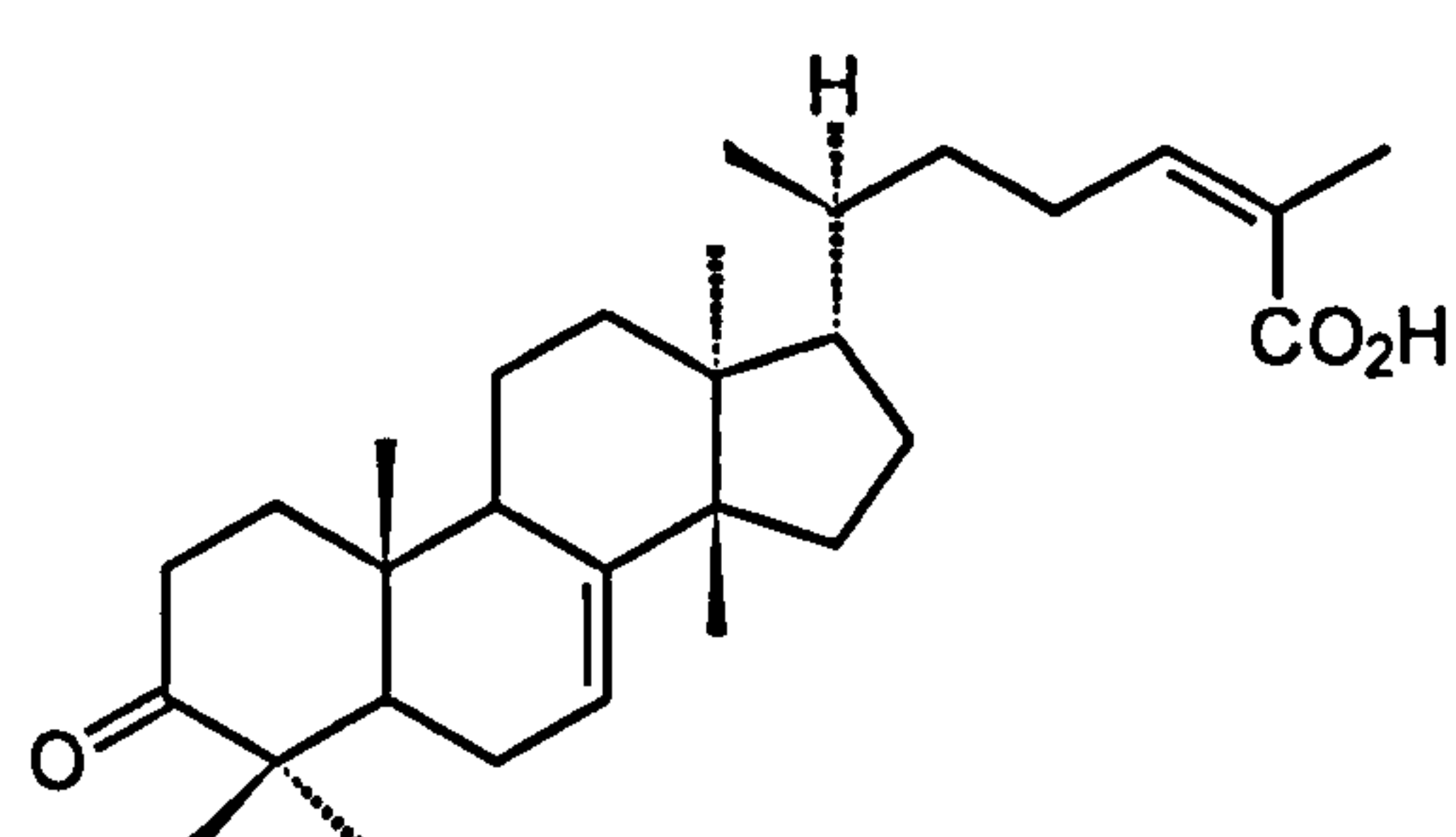
moronic acid



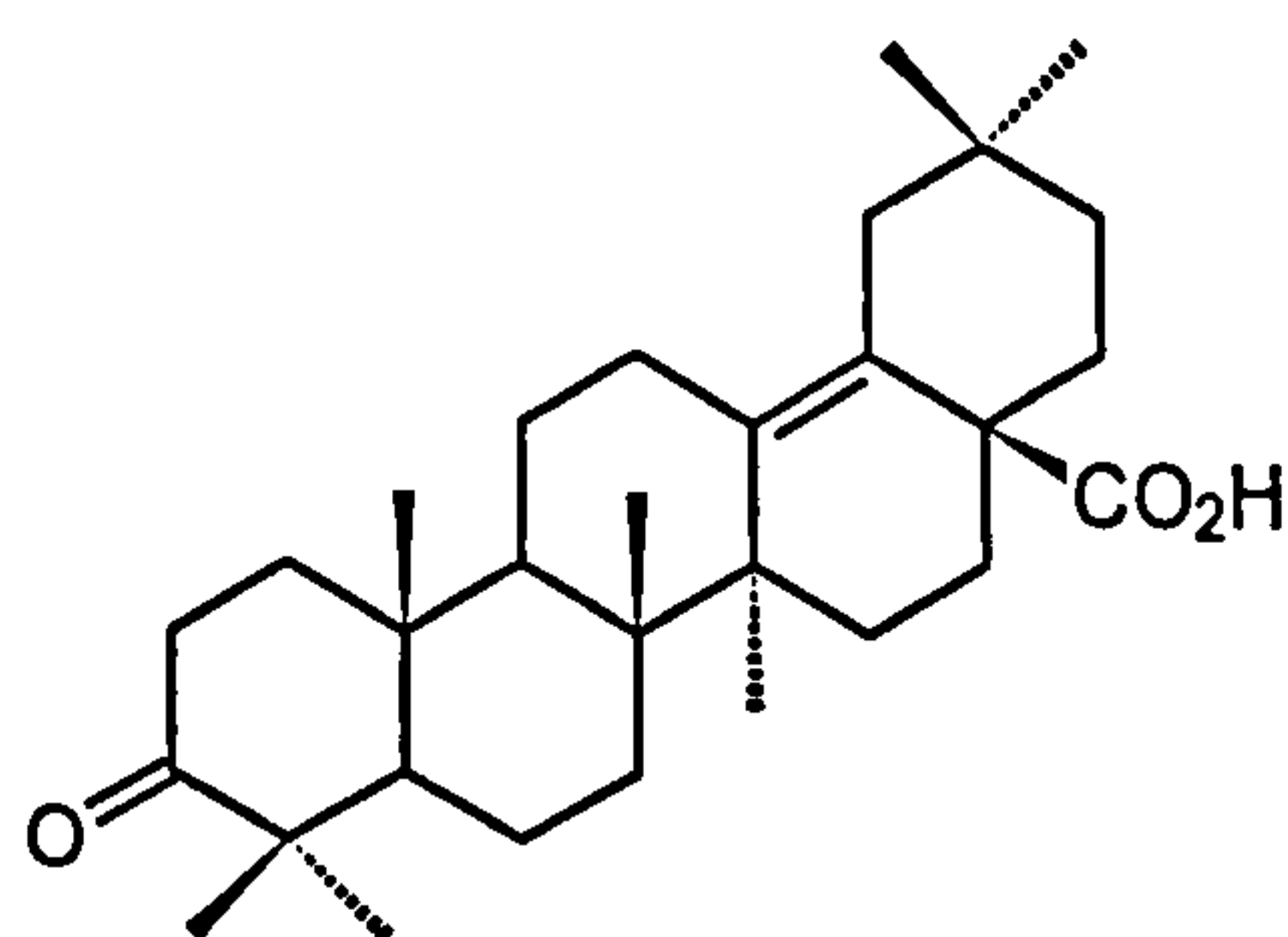
oleanonic acid



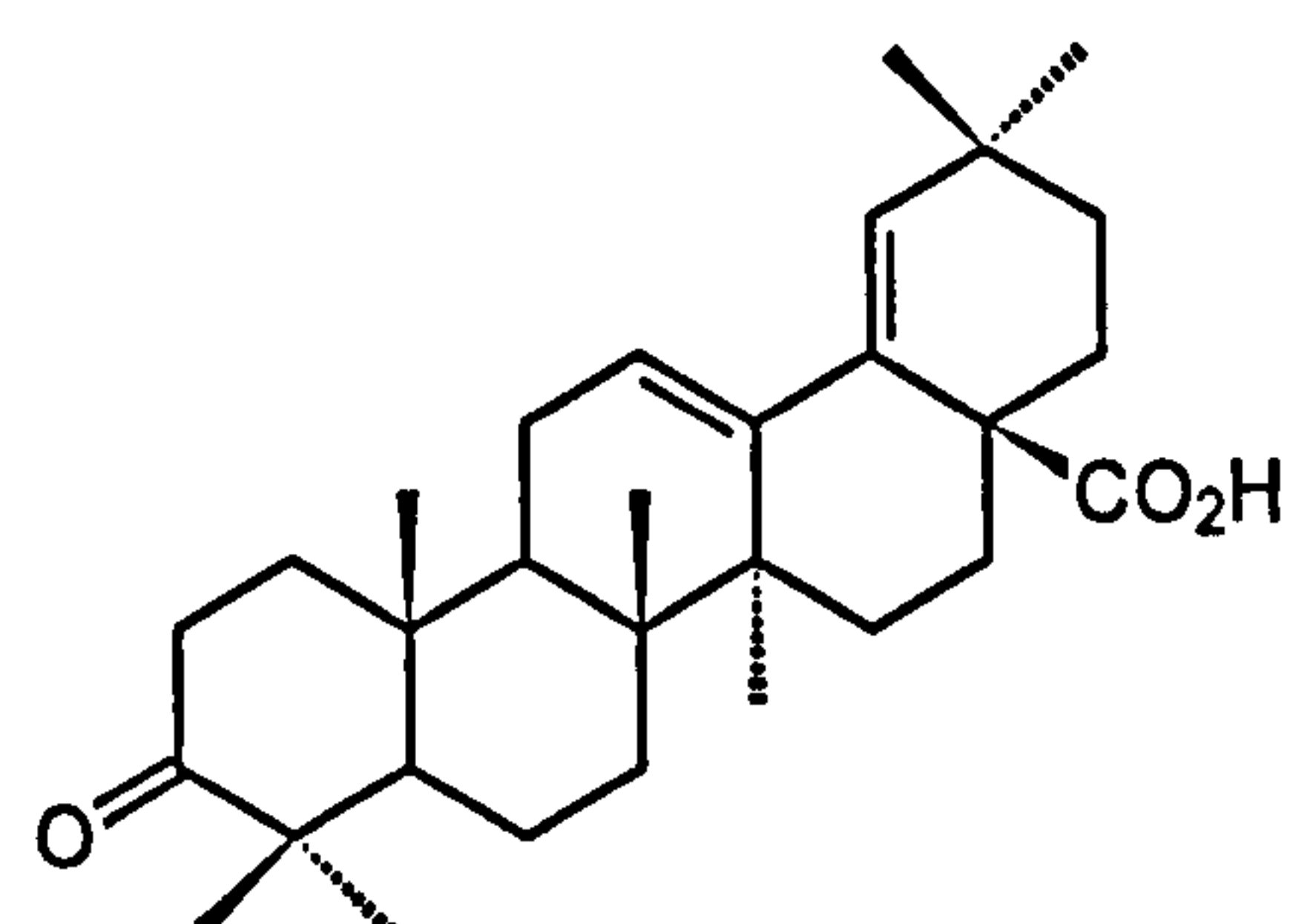
isomasticadienonic acid



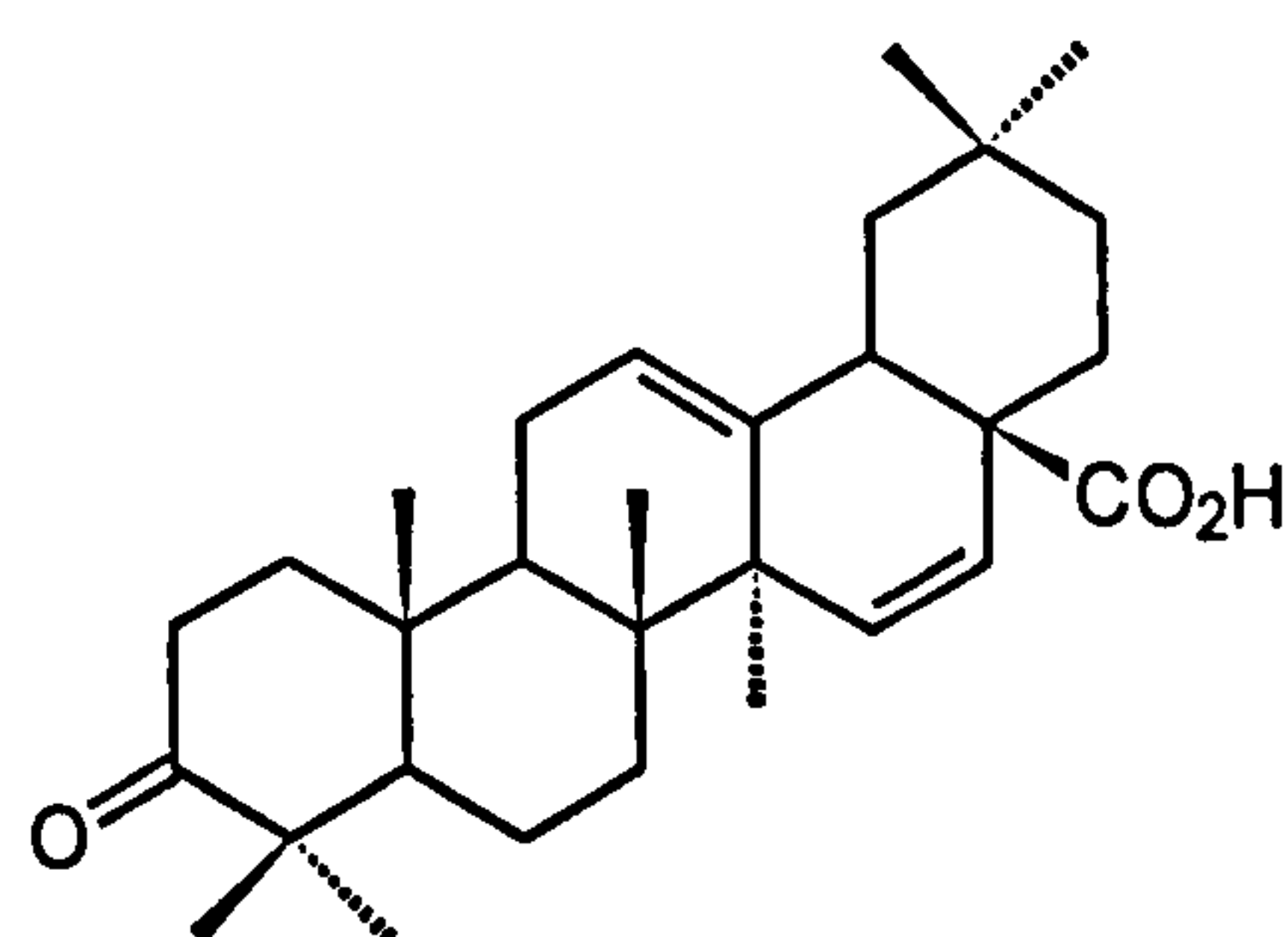
masticadienonic acid



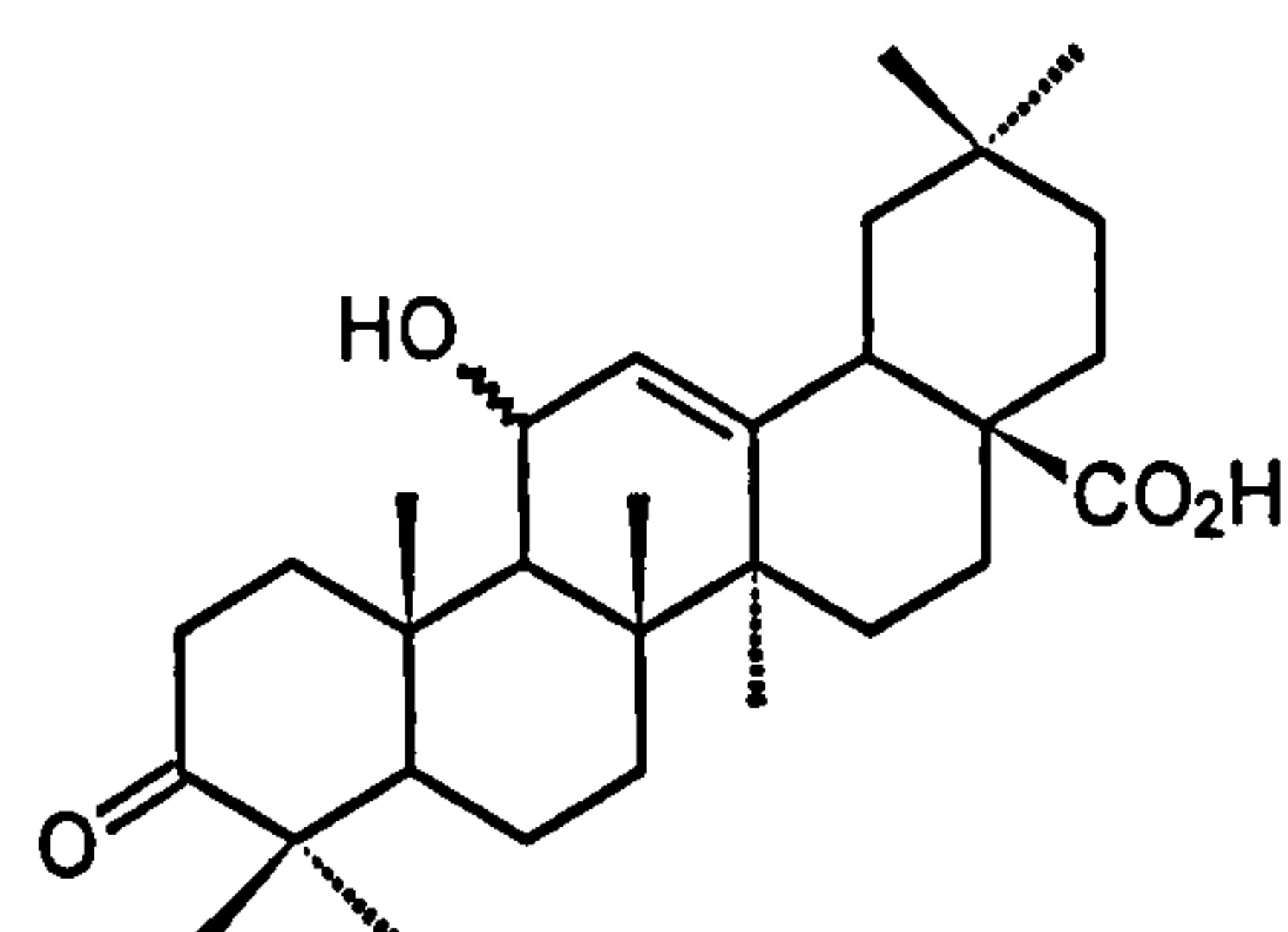
3-oxoolean-13-en-28-oic acid



3-oxoolean-12,18-dien-28-oic acid



3-oxoolean-12,15-dien-28-oic acid



11-hydroxyoleanonic acid

Figure 5.15 Triterpenoid acids from the 'resin' attached to linen thread from the right ankle/talus [1] of the Ptolemaic female adult (332-30B.C.)

3-oxoolean-12,18(?)-dien-28-oic acid, (m/z 73, 187, 239, 406 and M^+ 524), 3-oxoolean-12,15(?)-dien-28-oic acid (m/z 73, 187, 407 (base peak) and M^+ 524) and 11-hydroxyoleanonic acid (m/z 73, 161, 189, 203, 279, 309 and 427) (see Fig. 5.15). Due to the low abundance of these triterpenoid acids, the mass spectra obtained necessarily mean that these assignments can only be tentative. Nevertheless, the presence of moronic acid and these polyfunctionalised triterpenoids confirm an oxidised Pistacia resin as the main constituent of the acid fraction.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.14b. A series of *n*-alkanes (C_{23} to C_{33} , maximising at C_{27}) with an odd-over-even preference were observed, together with wax esters in the C_{38} to C_{60} carbon number range, C_{40} and C_{46} predominating. The C_{40} to C_{48} wax esters (even carbon numbers) contained the $C_{16:0}$ acyl group almost exclusively, in contrast to the C_{52} to C_{60} wax esters (even carbon numbers) containing mixtures of $C_{24:0}$ to $C_{30:0}$ acyl groups (even carbon numbers), increasing in chain length with an increase in wax ester carbon number (see discussion). The C_{50} wax ester contained $C_{16:0}$, $C_{18:0}$, $C_{24:0}$ and $C_{26:0}$ acyl groups, suggesting two different sources for this wax ester (see discussion below). Monohydroxy wax esters (C_{42} to C_{54}) were present as minor components, characterised by m/z 117 and 329, and eluting just after the wax esters of corresponding carbon number. Saturated fatty acid methyl esters ($C_{14:0}$ to $C_{34:0}$) were also observed in appreciable abundance, with $C_{16:0}$ and $C_{24:0}$ predominating – the distribution is consistent with the dominant acyl groups present in the wax esters. For their likely origin in the sample see 5.4.8.2 ‘Neutral fraction’. Other fatty acid methyl esters identified, albeit as minor components, were $C_{16:0}$ 5-oxo- and $C_{16:0}$ 5-hydroxy FAMES (see Figure 5.14). Minor quantities of C_{24} to C_{28} *n*-alkanols and two unidentified triterpenoids were also present. The neutral fraction constituted 94% of the total solvent extractable material.

5.4.9.2 ‘Resin’ from top of cranium [2].

TD-GC/MS

The results of the TD-GC/MS analysis displayed a series of *n*-alkanes (C_{23} to C_{33} with C_{27} predominating) and $C_{16:0}$ fatty acid, with 28-norolean-17-en-3-one also in significant abundance. The profile was very similar to the previous sample from this mummy (5.4.9.1).

Py-GC/MS

The results of the Py-GC/MS analysis displayed a series of alkene/alkane doublets (C₉ to C₂₃), with longer chain alkenes also dominant (C₂₄ to C₃₂). The fatty acid C_{16:0} is also significant, with 28-norolean-17-en-3-one present as a minor component. The pyrogram is similar to that obtained for the previous sample analysed from this mummy (5.4.9.1).

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS revealed aromatic acids identified as 4(?)-hydroxybenzoic, vanillic and protocatechuic acids. Also present were a series of saturated monocarboxylic acids (C_{16:0} to C_{28:0}) with C_{16:0} and C_{24:0} predominating. Triterpenoid acids were observed in significant abundance, the major triterpenoid being moronic, along with lesser amounts of oleanonic, 11-oxooleanonic, isomasticadienonic and masticadienonic acids, and the tentatively identified 3-oxoolean-12,18(?)-dien-28-oic, and 11-hydroxyoleanonic acid.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS revealed a series of *n*-alkanes (C₂₃ to C₃₃, maximising at C₂₇) with an odd-over-even preference, and wax esters in the C₃₈ to C₆₀ carbon number range, C₄₀ and C₄₆ predominating. The C₄₀ to C₄₈ wax esters (even carbon numbers) contained almost exclusively the C_{16:0} acyl group, whilst in contrast the C₅₂ to C₆₀ wax esters (even carbon numbers) contained mixtures of C_{24:0} to C_{30:0} acyl groups (even carbon numbers). The C₅₀ wax ester contained a mixture of acyl groups (C₁₆ to C_{26:0}) indicative of two different sources for the wax ester (see discussion below). Monohydroxy wax esters C₄₂ to C₅₄ were present as minor components (*m/z* 117 and 329) as was an unidentified triterpenoid, with saturated fatty acid methyl esters (C_{16:0} and C_{22:0}) in moderate abundance. The neutral fraction constituted 91% of the total solvent extractable material.

5.4.10 Male Adult, Roman (c.30 BC-AD 395), Hawara ('Portrait', 1911.2101)

5.4.10.1 'Resin'-soaked outer wrapping below right scapula [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.16a. A series of monocarboxylic acids (C₁₄ to C₁₈) were detected, the major components being C_{16:0}, C_{18:0}, C_{18:1} and C_{14:0} in decreasing order of abundance. Two C_{18:2} fatty acids were also identified

as significant constituents. Relatively minor components included the C₂₇ and C₂₉ *n*-alkanes and the oxidised diterpenoids 7-oxo-18-norabieta-3,5,8,11,13-pentaene, 7-oxo-18-norabieta-3,5,8,11,13,15-hexaene and methyl 7-oxodehydroabietate.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.16a, inset. The pyrogram revealed the monocarboxylic acids C_{16:0}, C_{18:2}, C_{18:1} and C_{18:0} as major components. Levoglucosan was also abundant, as were furan and pyran derivatives, although given the origin of the sample, i.e. cellulose-base wrappings, this is not unexpected.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.16b, inset. A series of monocarboxylic acids (C₉ to C₂₈) were identified, with C_{16:0}, C_{18:0}, C_{18:1} and C_{14:0} predominant in decreasing amounts of abundance. The distribution of these acids was similar to that obtained by TD. Also present in significant amounts were a series of α,ω -dicarboxylic acids (C₄ to C₁₁), in addition to C_{18:1} monohydroxy- and C_{18:0} 9,10-dihydroxy carboxylic acids. Diterpenoids were observed as significant components, with appreciable quantities of dehydroabietic, 7-oxo-dehydroabietic and 15-hydroxy-7-oxodehydroabietic acids. A trace of hydrocinnamic acid was also detected.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.16b. A series of *n*-alkanes (C₂₅ to C₃₃, maximising at C₂₇) with an odd-over-even preference were observed, as were wax esters (C₄₀ to C₅₀, maximising at C₄₆). The C_{16:0} acyl group was predominant in these wax esters, with smaller amounts of C_{18:0}. Minor amounts of fatty acid methyl esters (C₁₆ to C₁₈) were also detected. The neutral fraction constituted a significant, but relatively small, proportion of the total extract (10%).

5.4.10.2 'Resin'-soaked wrapping from front at base of right side of thorax [3].

TD-GC/MS

The results of the TD-GC/MS analysis displayed a series of monocarboxylic acids (C₁₄ to C₁₈), the major components being C_{16:0}, C_{18:0}, C_{18:1} and C_{14:0} in decreasing order of abundance. Two C_{18:2} fatty acids were also identified as major constituents. Other components identified included the C₂₇ and C₂₉ *n*-alkanes and a number of oxidised

diterpenoids, among which were 7-oxo-18-norabieta-3,5,8,11,13-pentaene, 7-oxo-18-norabieta-3,5,8,11,13,15-hexaene and methyl 7-oxodehydroabietate.

Py-GC/MS

The results of the Py-GC/MS analysis revealed the monocarboxylic acids $C_{16:0}$, $C_{18:2}$, $C_{18:1}$ and $C_{18:0}$ as significant components in decreasing order of abundance. Levoglucosan was also observed in appreciable quantity, with significant amounts of furan and pyran derivatives, although given the origin of the sample, i.e. cellulose-base wrapping, this is not unexpected.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS revealed a series of monocarboxylic acids (C_{14} to C_{24}), with $C_{16:0}$, $C_{18:0}$, $C_{18:1}$ and $C_{14:0}$ predominating in decreasing order of abundance. The distribution of these acids was similar to that obtained by TD. Also present in significant amounts were a series of α,ω -dicarboxylic acids (C_8 to C_{10}), in addition to $C_{18:1}$ monohydroxy- and $C_{18:0}$ 9,10-dihydroxycarboxylic acids. Diterpenoids were observed as significant components, with appreciable quantities of dehydroabietic, 7-oxo-dehydroabietic and 15-hydroxy-7-oxodehydroabietic acids.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS displayed no significant neutral components, the chromatogram being dominated by the internal standard (C_{34} *n*-alkane).

5.4.11 Male Adult, Roman (c.30 BC-AD 395), Hawara ('Mask', 1911.2102)

5.4.11.1 'Resin' from side/base of left foot [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.17a. The monocarboxylic acids $C_{16:0}$ and $C_{18:0}$ were the major components, present in equal abundance. The diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate were observed in moderate abundance, with more significant quantities of decarboxylated 7-oxo-diterpenoids. Further major components were a series of *n*-alkanes, C_{25} to C_{31} , with an odd-over-even preference.

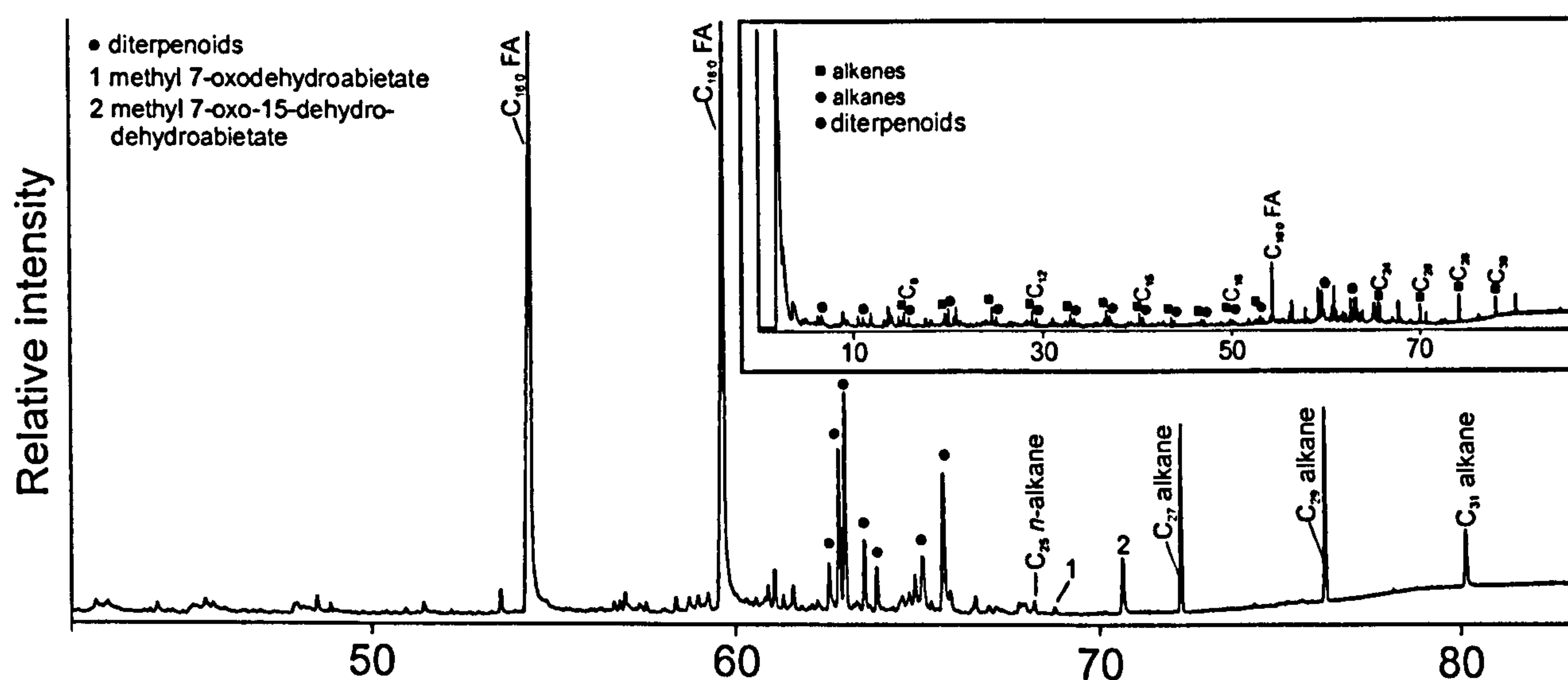


Figure 5.17a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of ‘resin’ from the side/base of the left foot [1] of the Roman Period male adult ‘mask’, (c. 30 B.C. -395 A.D.).

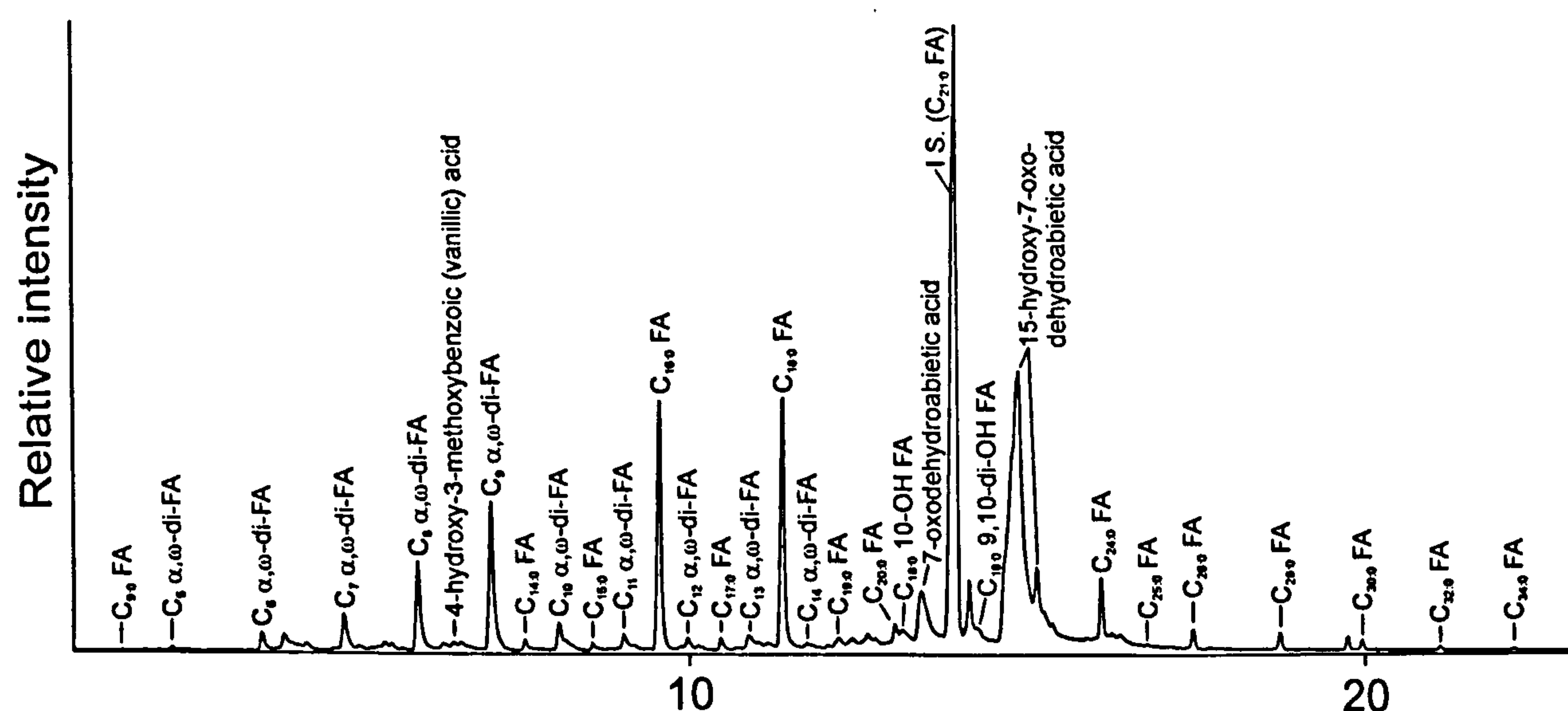


Figure 5.17b Total ion chromatogram from the GC/MS analysis of the acid fraction of ‘resin’ from the side/base of the left foot [1] of the Roman Period male adult ‘mask’, (c. 30 B.C. -395 A.D.).

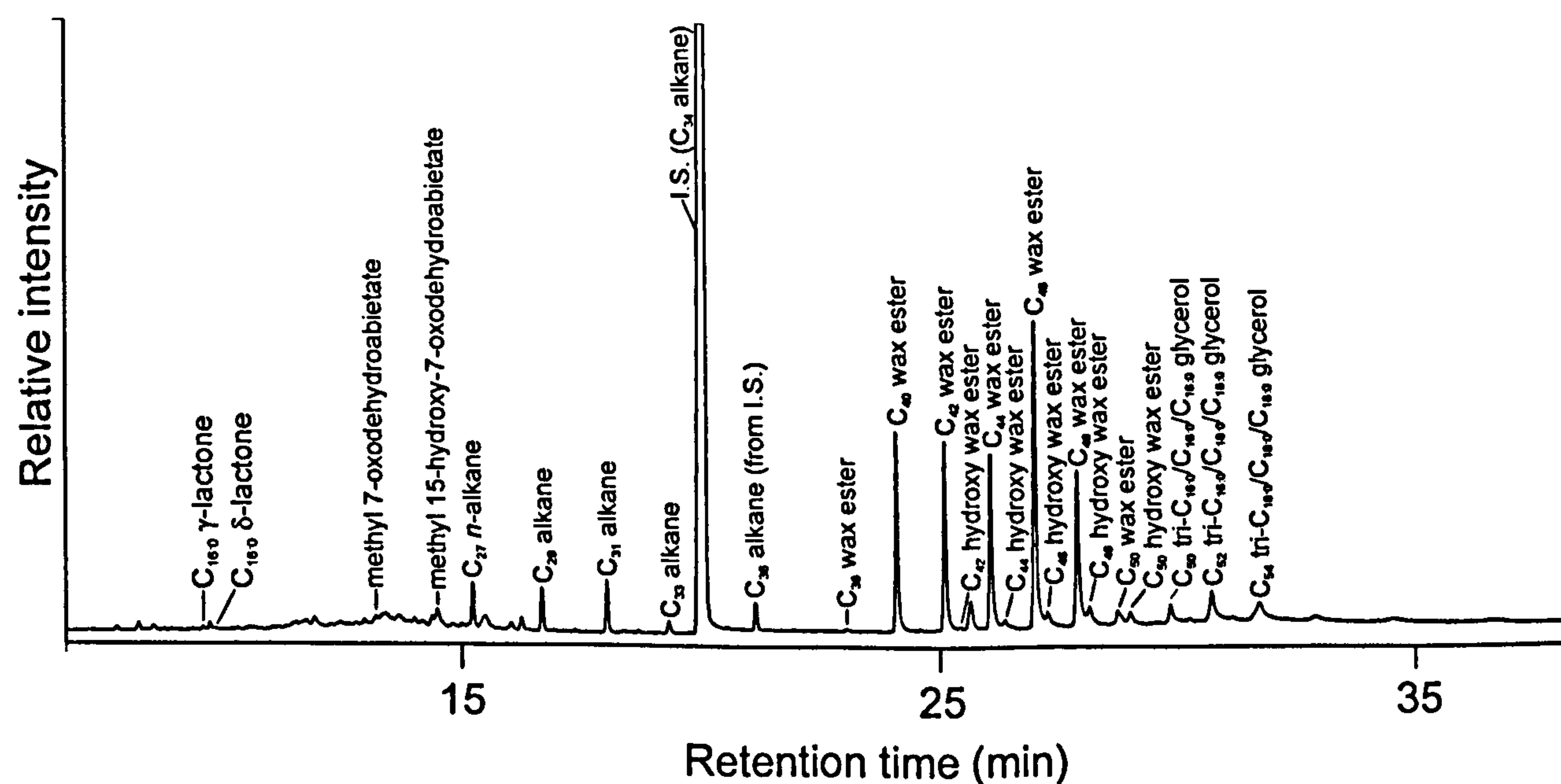


Figure 5.17c Total ion chromatograms from the GC/MS analysis of the neutral fraction of ‘resin’ from the side/base of the left foot [1] of the Roman Period male adult ‘mask’, (c. 30 B.C. -395 A.D.).

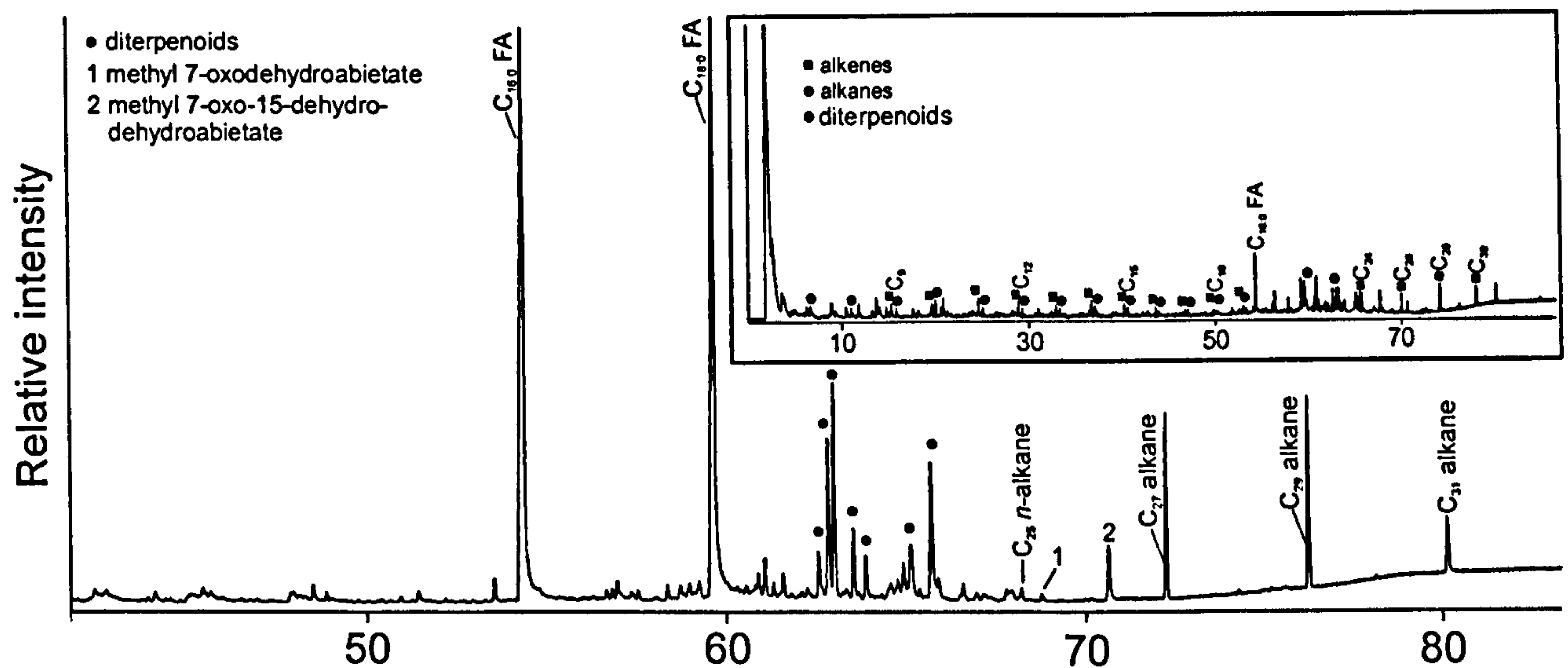


Figure 5.17a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of ‘resin’ from the side/base of the left foot [1] of the Roman Period male adult ‘mask’, (c. 30 B.C. -395 A.D.).

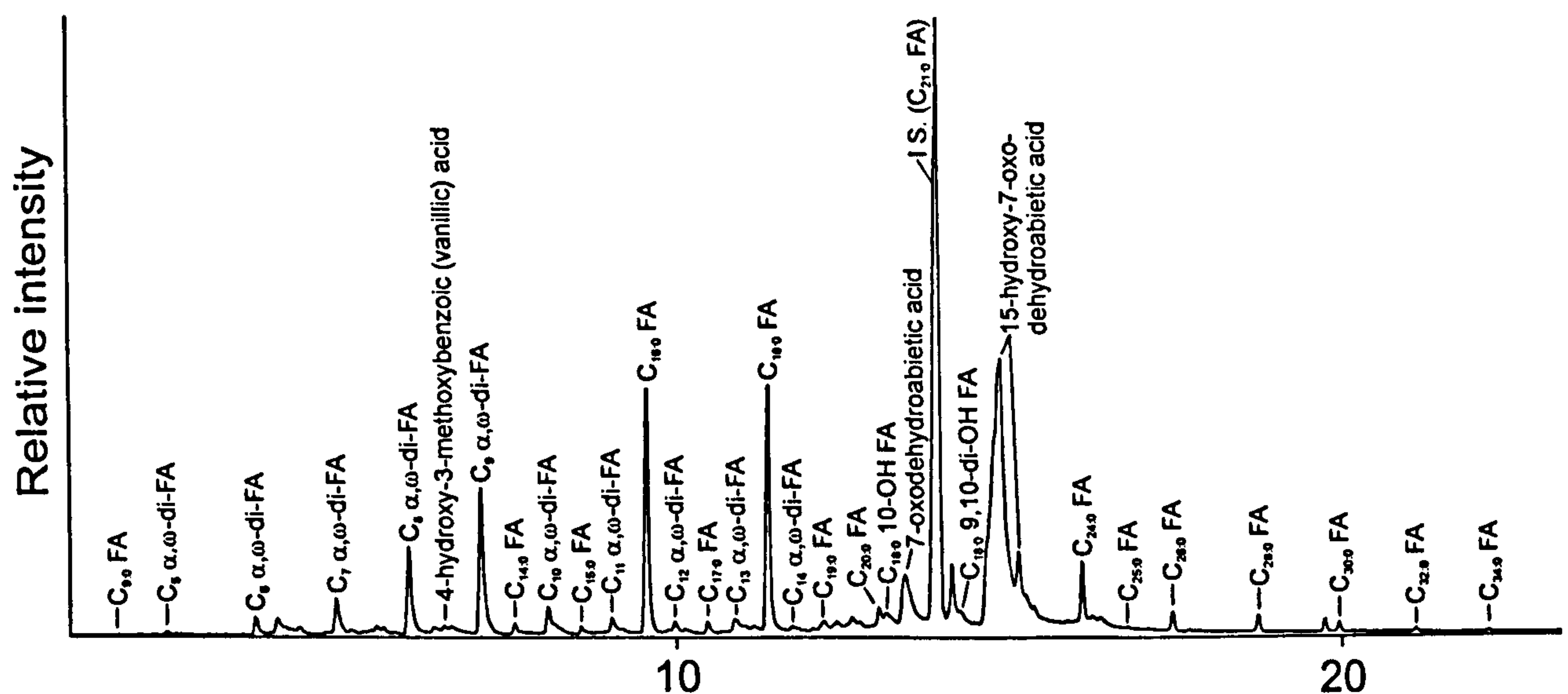


Figure 5.17b Total ion chromatogram from the GC/MS analysis of the acid fraction of 'resin' from the side/base of the left foot [1] of the Roman Period male adult 'mask', (c. 30 B.C. -395 A.D.).

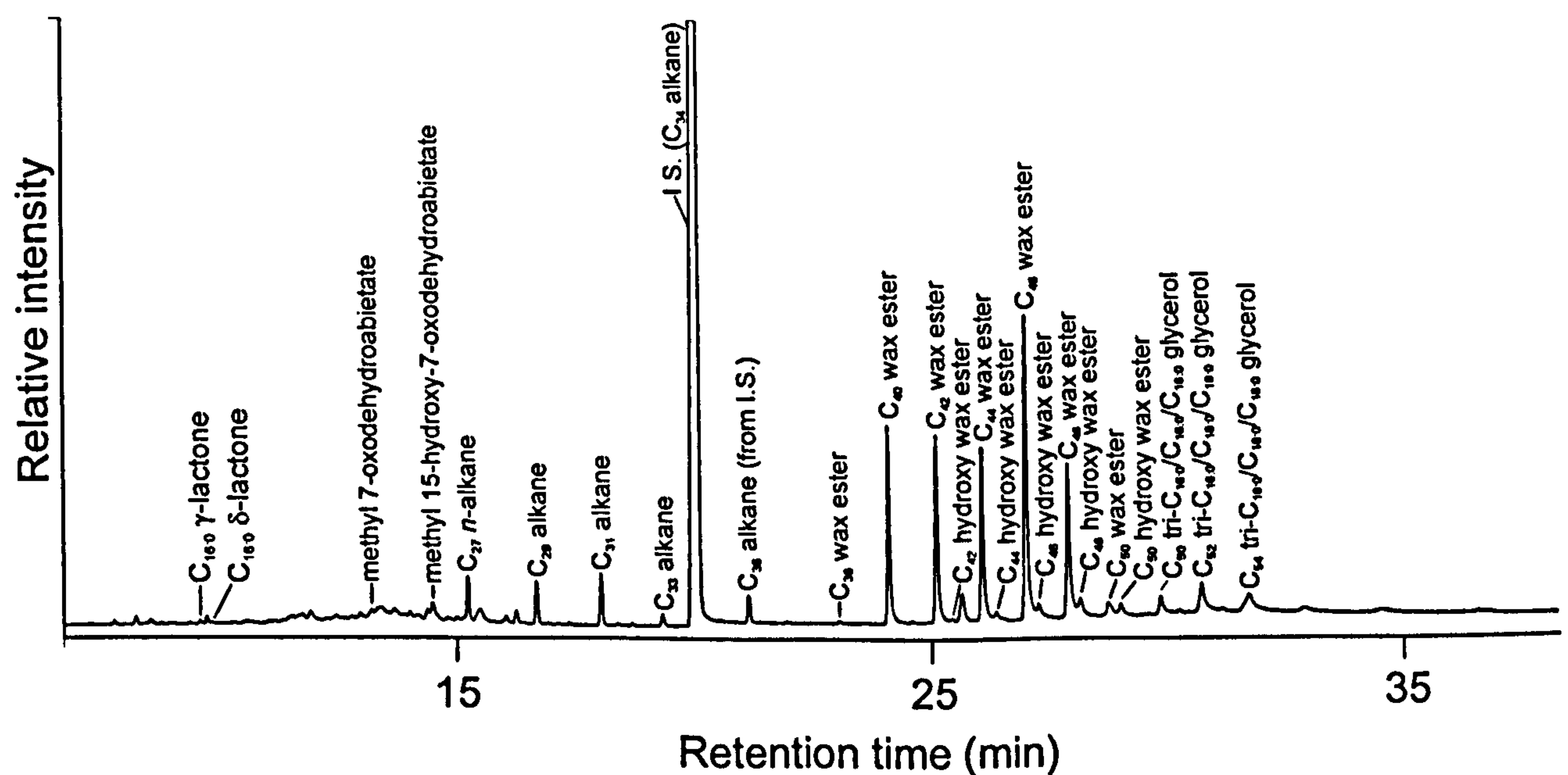


Figure 5.17c Total ion chromatograms from the GC/MS analysis of the neutral fraction of ‘resin’ from the side/base of the left foot [1] of the Roman Period male adult ‘mask’, (c. 30 B.C. -395 A.D.).

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.17a, inset. The pyrogram revealed the monocarboxylic acids C_{16:0} and C_{18:0} as major components (for their likely origin see discussion) The pyrogram is dominated by alkene/alkane doublets (C₈ to C₂₃) maximising *n*-C₉ to C₁₁, with longer chain alkenes also significant (C₂₄ to C₃₀) (for the likely origin of these see discussion below). Appreciable abundances of defunctionalised diterpenoids, including the decarboxylated 7-oxo-abietanes, were also identified in addition to minor quantities of methyl 7-oxo-dehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.17b. A series of saturated monocarboxylic acids (C_{9:0} to C_{34:0}) were identified, with C_{16:0} and C_{18:0} predominating in equal abundance. Long chain fatty acids (C_{24:0} to C_{34:0}) were detected in appreciable amounts, maximising at C_{24:0}. The distribution of the monocarboxylic acids was similar to that obtained by TD, although due to their increasing involatility the longer chain acids were more abundance in the acid fraction than in the TD profile. Also present in significant amounts were a series of α,ω -dicarboxylic acids (C₅ to C₁₄), with suberic and azelaic the major of these. Other lipid oxidation products, i.e. C_{18:1} monohydroxy- and C_{18:0} 9,10-dihydroxy-carboxylic acids were detected as minor components. Oxidised diterpenoids were observed, with appreciable quantities of 7-oxo-dehydroabietic acid and major quantities of 15-hydroxy-7-oxodehydroabietic acids (the major compound of the acid fraction and indeed the whole sample), with dehydroabietic acid notably absent. A trace of vanillic acid was also detected.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.17c. A series of *n*-alkanes (C₂₇ to C₃₃) with an odd-over-even preference were observed, together with wax esters in the C₄₀ to C₅₀ carbon number range, C₄₀ and C₄₆ predominating. The C_{16:0} acyl group was predominant in these wax esters, with only minor amounts of C_{18:0} in all but the C₅₀ ester. Monohydroxy was esters (C₄₂ to C₅₄) were present as minor components, characterised by *m/z* 117 and 329, and eluting just after the wax esters of corresponding carbon number. Eluting just after the wax esters, three triacylglycerols C₅₀ tri-C_{16:0}/C_{16:0}/C_{18:0}-glycerol, C₅₂ tri-C_{16:0}/C_{18:0}/C_{18:0}-glycerol and C₅₄ tri-C_{18:0}/C_{18:0}/C_{18:0}-glycerol were observed as significant components. Minor quantities of methyl 7-

oxodehydroabietate and methyl 15-hydroxy-7-oxodehydroabietate and the C_{16:0} γ - and δ -lactones were also detected. The neutral fraction constituted a significant amount (27%) of the total solvent extractable material.

5.4.11.2 'Resin' from left strip of wrapping at base of golden mask [3].

TD-GC/MS

The results of the TD-GC/MS analysis displayed the monocarboxylic acids C_{16:0} and C_{18:0} were the major components, present in similar abundance. The diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate were observed in moderate abundance, with more significant quantities of decarboxylated 7-oxo-diterpenoids. Further major components were a series of *n*-alkanes, C₂₅ to C₃₁, with an odd-over-even preference.

Py-GC/MS

The results of the Py-GC/MS analysis revealed the monocarboxylic acids C_{16:0} and C_{18:0} as significant components (for their likely origin see discussion) together with appreciable abundances of defunctionalised diterpenoids, including the decarboxylated 7-oxo-abietanes, were also identified. Levoglucosan was detected, although given that the sample originated from cellulose-base wrapping, this is not unexpected.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS revealed a series of saturated monocarboxylic acids (C_{14:0} to C_{32:0}) were identified, with C_{16:0} and C_{18:0} predominating in equal abundance. Long chain fatty acids (C_{24:0} to C_{32:0}) were detected in moderate amounts, maximising at C_{24:0}. The distribution of the monocarboxylic acids was similar to that obtained by TD, although due to their increasing involatility the longer chain acids were more abundance in the acid fraction than in the TD profile. Also present in significant amounts were a series of α,ω -dicarboxylic acids (C₆ to C₁₄), with suberic and azelaic the major of these. Other lipid oxidation products, i.e C_{18:1} monohydroxy- and C_{18:0} 9,10-dihydroxy-carboxylic acids were detected as minor components. Oxidised diterpenoids were observed, with appreciable quantities of 7-oxo-dehydroabietic acid and major quantities of 15-hydroxy-7-oxodehydroabietic acids, with dehydroabietic acid notably absent. A trace of vanillic acid was also detected.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS displayed a series of *n*-alkanes (C₂₇ to C₃₃) with an odd-over-even preference were observed, together with wax esters in the C₄₀ to C₅₀ carbon number range, C₄₀ and C₄₆ predominating. The C_{16:0} acyl group was predominant in these wax esters, with only minor amounts of C_{18:0} in all but the C₅₀ ester. Monohydroxy wax esters (C₄₂ to C₅₄) were present as minor components, characterised by *m/z* 117 and 329, and eluting just after the wax esters of corresponding carbon number. Eluting just after the wax esters, three triacylglycerols C₅₀ tri-C_{16:0}/C_{16:0}/C_{18:0}-glycerol, C₅₂ tri-C_{16:0}/C_{18:0}/C_{18:0}-glycerol and C₅₄ tri-C_{18:0}/C_{18:0}/C_{18:0}-glycerol were observed as significant components. The neutral fraction constituted a third (33%) of the total solvent extractable material.

5.4.12 Male Child, Roman (c.30 BC-AD 395), Thebes ('wrapped twin', 1956.357b)*5.4.12.1 'Resin' on outer wrapping from area between calves [1].*

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.18a. A series of monocarboxylic acids (C₁₂ to C₁₈) were detected, the major components being C_{16:0}, C_{18:1}, C_{18:0} and C_{14:0} in decreasing order of abundance. Two C_{18:2} fatty acids were also identified as significant constituents along with the oxidised decarboxylated diterpenoids 7-oxo-18-norabieta-3,5,8,11,13-pentaene, 7-oxo-18-norabieta-3,5,8,11,13,15-hexaene. Levoglucosan was identified as a significant component, possibly indicative of a gum/sugar origin.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.18.a, inset. The pyrogram revealed the monocarboxylic acids C_{16:1}, C_{16:0}, C_{18:1}, C_{18:0} and C_{14:0} as major components along with two C_{18:2} fatty acids. A series of alkene/alkane doublets (C₈ to C₁₅) were also present in significant abundance. Levoglucosan, observed in the TD, was absent supporting the suggestion of its gum/sugar origin, particularly given the absence of cellulose-based wrappings in this sample.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.18b. A series of monocarboxylic acids (C₁₂ to C₂₄) were identified, with C_{16:0}, C_{18:0}, C_{18:1} and C_{14:0} predominating in decreasing order of abundance. Moderate amounts of the mono-

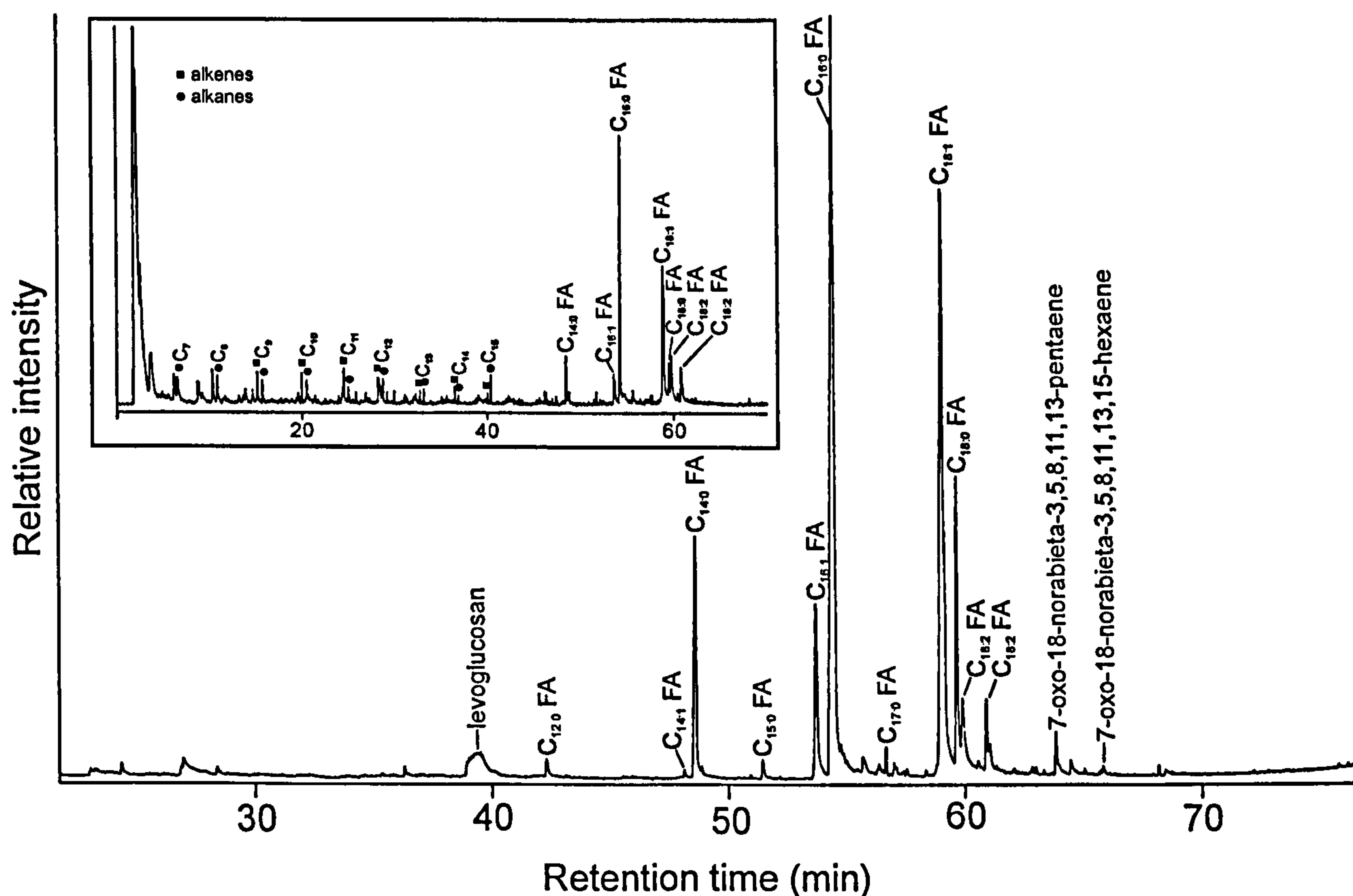


Figure 5.18a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin' on the outer wrapping from the area between the calves [1] of the Roman Period male child 'wrapped twin', (c. 30 B.C. -395 A.D.).

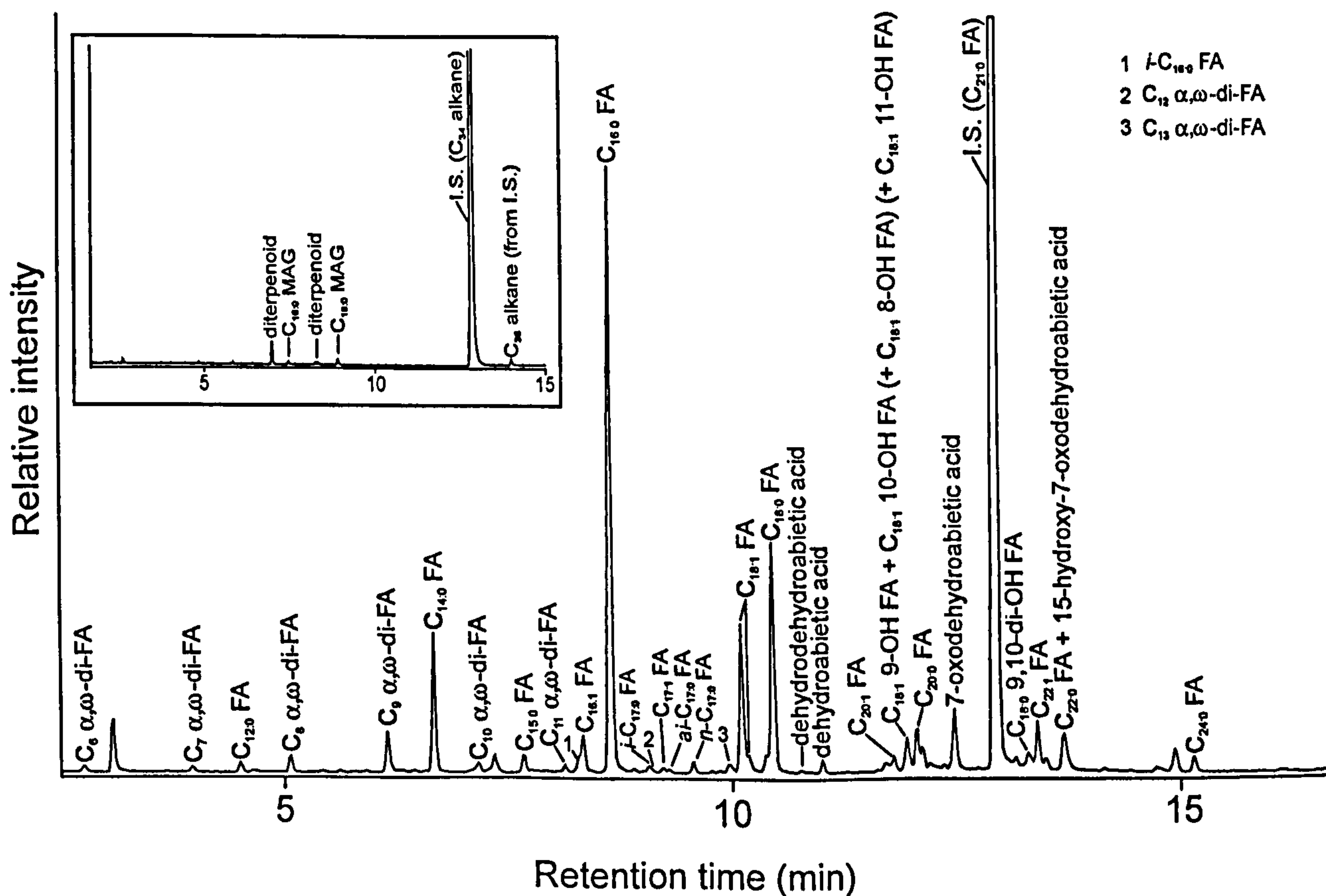


Figure 5.18b Total ion chromatograms from the GC/MS analyses of the acid fraction, and (inset) neutral fraction of 'resin' on the outer wrapping from the area between the calves [1] of the Roman Period male child 'wrapped twin', (c. 30 B.C. -395 A.D.).

unsaturated fatty acids C_{16:1}, C_{20:1} and C_{22:1} were also present. A series of α,ω -dicarboxylic acids (C₆ to C₁₃), and C_{18:1} monohydroxy- and C_{18:0} 9,10-dihydroxy-carboxylic acids were detected in appreciable abundance. Dehydrodehydroabietic and dehydroabietic acid were present as minor components, with more appreciable quantities of 7-oxodehydroabietic and 15-hydroxy-7-oxodehydroabietic acids.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.18b, inset. Minor quantities of the C_{16:0} and C_{18:0} 1-monoacyl-glycerols and two unidentified diterpenoids were the only neutral components detected.

5.4.12.2 'Resin' stained wrapping from back of mummy at base of thorax [2].

TD-GC/MS

The results of the TD-GC/MS analysis displayed a series of monocarboxylic acids (C₁₂ to C₁₈) were detected, the major components being C_{16:0}, C_{18:1}, C_{18:0} and C_{14:0} in decreasing order of abundance with defunctionalised diterpenoids also in significant abundance.

Py-GC/MS

Py-GC/MS analysis revealed the monocarboxylic acids C_{16:0}, C_{18:1}, C_{18:0} and C_{14:0} as significant components with similar amounts of defunctionalised diterpenoids. Levoglucosan was also abundant, together with furan and pyran derivatives, although given the origin of the sample i.e. cellulose-based wrappings, this is not unexpected.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS displayed a series of monocarboxylic acids (C₁₄ to C₂₂) were identified, with C_{16:0}, and C_{18:0} predominating. Minor amounts of the monounsaturated fatty acids C_{16:1} and C_{18:1} were also present. Dehydroabietic and 15-hydroxy-7-oxodehydroabietic acids were present as minor components, with more appreciable quantities of 7-oxodehydroabietic acid.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS displayed no significant neutral components. Trace quantities of the C_{16:0} and C_{18:0} 1-monoacyl-glycerols and two unidentified diterpenoids were the only neutral components detected, the chromatogram being dominated by the internal standard (C₃₄ *n*-alkane).

5.4.13 Male Child, Roman (c.30 BC-AD 395), Thebes ('unwrapped twin', 1956.357c)

5.4.13.1 'Resin' from abdominal cavity (kidney area).

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 5.19a. A series of monocarboxylic acids (C_{12} to C_{18}) was detected, the major components being $C_{16:0}$, $C_{18:1}$, $C_{14:0}$ and $C_{18:0}$ in decreasing order of abundance. The oxidised diterpenoids 7-oxo-18-norabieta-3,5,8,11,13-pentaene and 7-oxo-18-norabieta-3,5,8,11,13,15-hexaene were present in moderate abundance with methyl 7-oxodehydroabietate identified as a minor constituent.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 5.19a, inset. The pyrogram revealed the monocarboxylic acids $C_{14:0}$, $C_{16:1}$, $C_{16:0}$, $C_{18:1}$, and $C_{18:0}$ as major components along with a series of alkene/alkane doublets (C_8 to C_{15}) present in significant abundance. An unidentified diterpenoid was the major component of the pyrolysis profile.

GC/MS-Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 5.19b. A series of monocarboxylic acids ($C_{12:0}$ to $C_{24:0}$) were identified, with $C_{16:0}$, $C_{18:1}$ and $C_{14:0}$ predominating in decreasing order of abundance. Significant amounts of the monounsaturated fatty acids $C_{14:1}$, $C_{16:1}$, $C_{17:1}$, $C_{20:1}$, $C_{22:1}$ and $C_{24:1}$ were also present. A series of α,ω -dicarboxylic acids (C_6 to C_{12}), were detected in moderate abundance with minor quantities of $C_{18:1}$ monohydroxy- and $C_{18:0}$ 9,10-dihydroxy-carboxylic acids. Dehydroabietic and 7-oxodehydroabietic acids were observed as significant constituents, with lesser amounts of Dehydrodehydroabietic and 15-hydroxy-7-oxodehydroabietic acids. Most notable however, was the detection of isopimaric acid, albeit as a minor component, this pimarane type diterpenoid only survives in favourable environments. Vanillic acid was also detected as a trace component.

GC/MS - Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 5.19c. Minor quantities of the $C_{12:0}$, $C_{14:0}$, $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ 1-monoacyl-glycerols were identified with (notably) the $C_{14:0}$ the most abundant of these. The two steroidal components

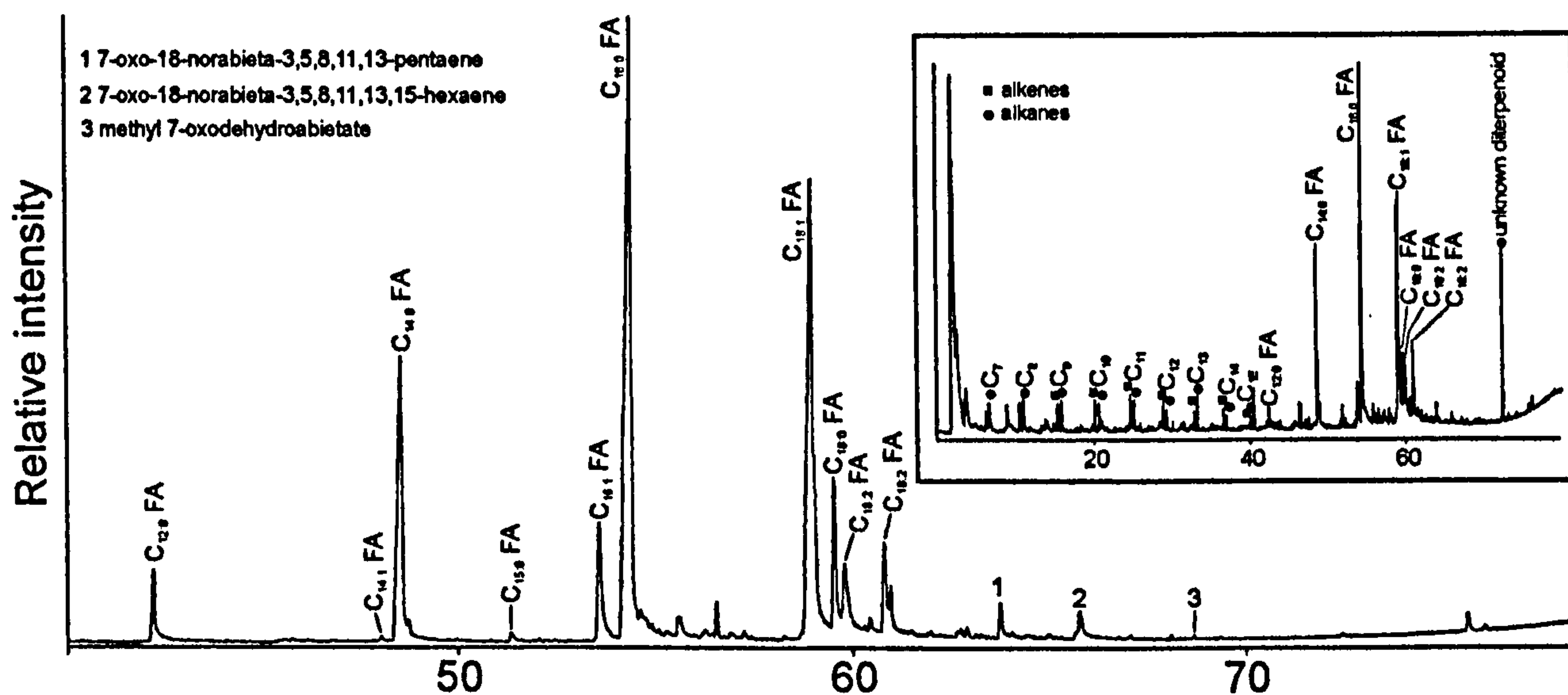


Figure 5.19a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of ‘resin’ from the abdominal cavity (kidney area) of the Roman Period male child ‘unwrapped twin’, (c. 30 B.C. -395 A.D.).

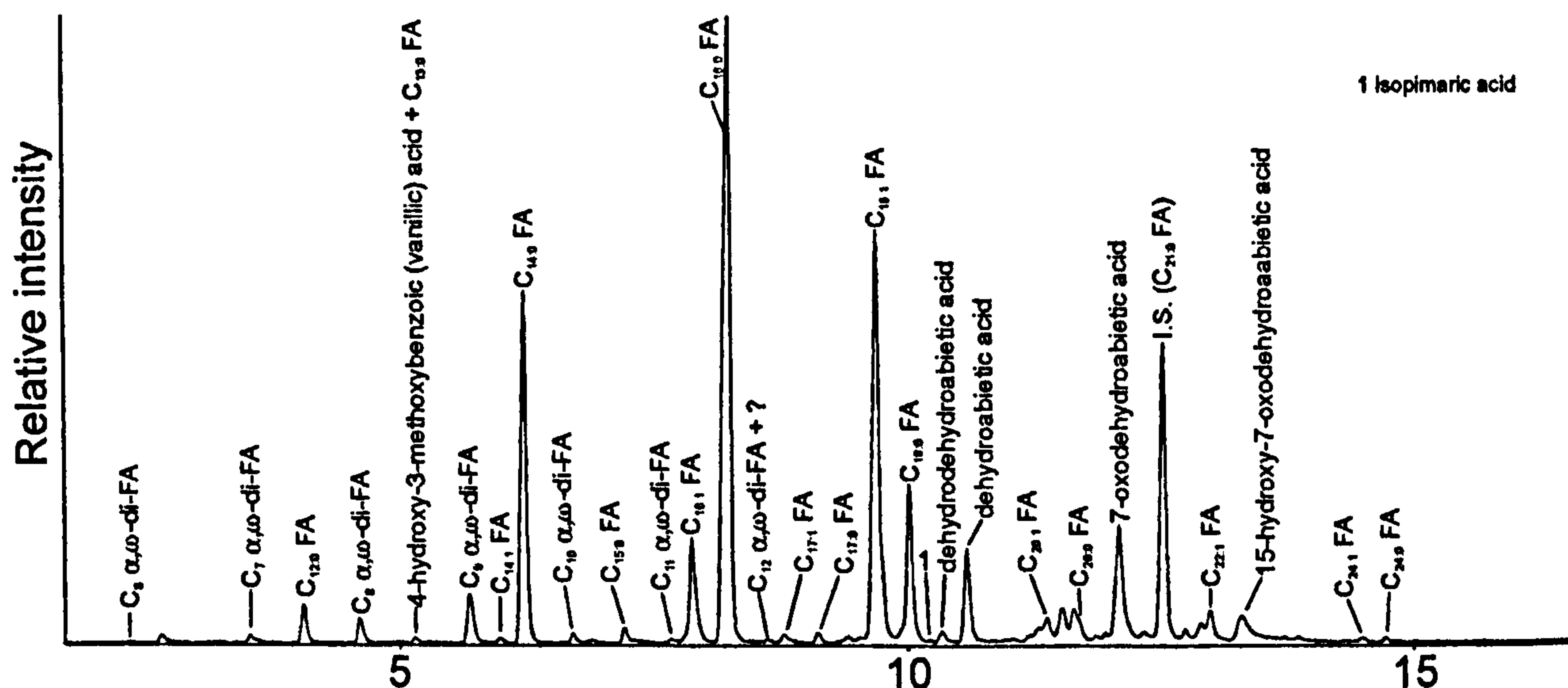


Figure 5.19b Total ion chromatogram from the GC/MS analysis of the acid fraction of 'resin' from the abdominal cavity (kidney area) of the Roman Period male child 'unwrapped twin', (c. 30 BC -395 AD)

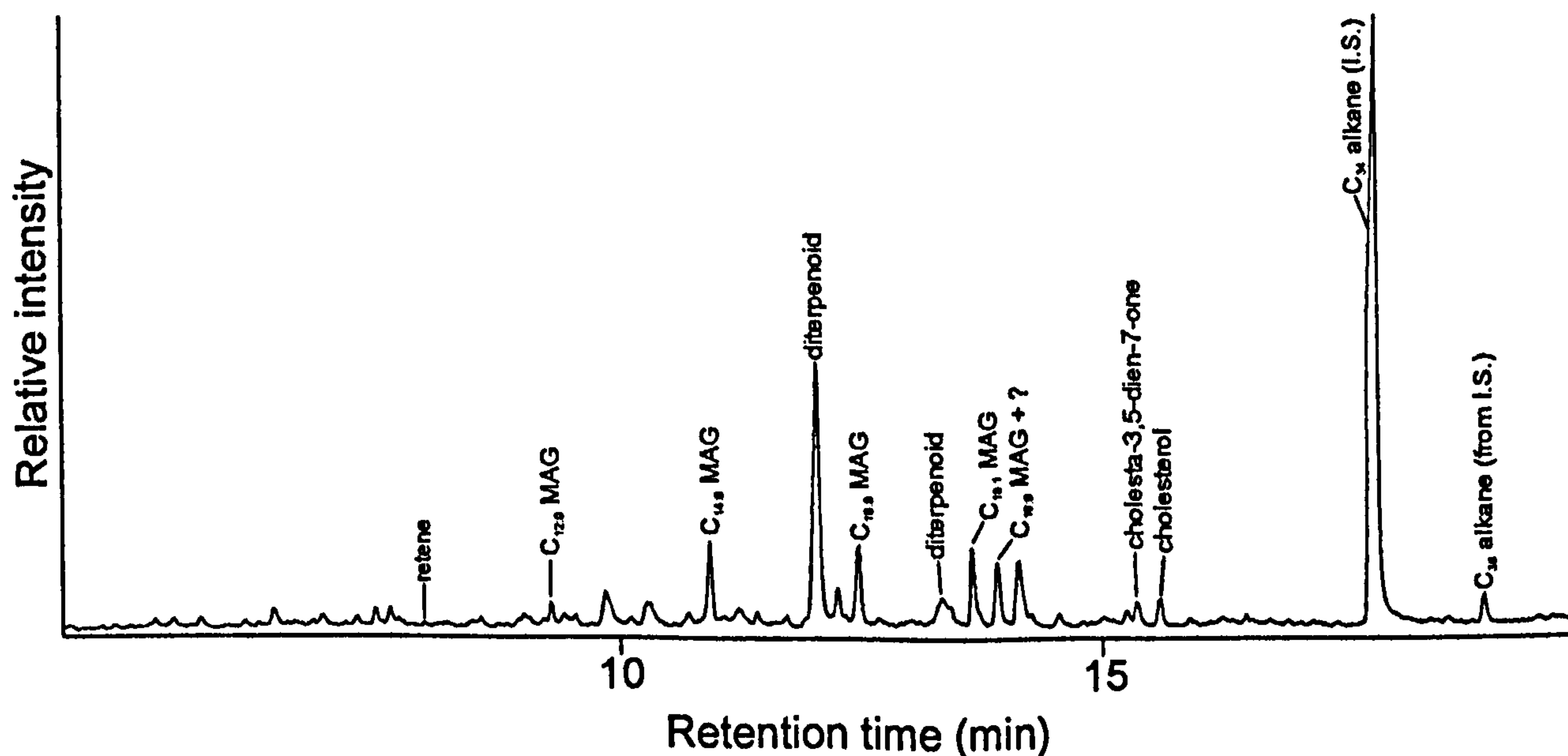


Figure 5.19c Total ion chromatogram from the GC/MS analysis of the neutral fraction of ‘resin’ from the abdominal cavity (kidney area) of the Roman Period male child ‘unwrapped twin’, (c. 30 B.C. -395 A.D.).

cholesta-3,5-dien-7-one and cholesterol were also minor components along with two unidentified diterpenoids.

It should be noted that mass chromatograms were obtained for all samples in order to ascertain whether diterpenoids (m/z 239, 251, 253), triterpenoids (m/z 163, 189, 203) and steranes (m/z 217) and hopanes (m/z 191) were present in trace amounts.

5.5 DISCUSSION

Analysis of the three samples taken from the Old Kingdom mummy (5.4.1) were very similar in composition with only minor differences. The lesser amounts of the longer chain *n*-alkanes in the TD in comparison to the solvent extract is no doubt due to the lower volatilities of these compounds, thus the more volatile, shorter chain *n*-alkanes are preferentially volatilised in the probe at 310°C, as supported by the increased abundance of the longer chain alkane at the higher pyrolysis temperature of 610°C. If the longer chain *n*-alkanes were added to the TD profile, the *n*-alkane profiles for both the TD-GC/MS analysis and the solvent extraction would be almost identical. Their distribution of hydrocarbons, dominated by the C₂₀ to C₄₀ *n*-alkanes, with no odd-over-even preference, and maximising between C₂₄ and C₂₆, clearly identified them as a paraffin wax. As a result of subsequent enquiries, museum records did in fact reveal that the mummy had been treated with paraffin wax following its excavation by Flinders Petrie in 1910, a period when the wax was commonly applied to mummies in an attempt to preserve them (as discussed above in Chapter 3). Although this may appear to be a negative result, it does serve to illustrate that visual appearance can in no way be used to identify the origin of these amorphous organic materials. The black and shiny nature of the samples from this mummy fits the all-too-often-used descriptions 'resin' or 'bitumen', illustrating how dangerously misleading it can be to use visual appearance to identify the so-called resins used – which in this particular case was paraffin wax!

The bandage/resin/tissue samples from the Middle Kingdom mummy 'Khnumnakht' (5.4.2) consisted largely of free fatty acids with appreciable amounts of cholesterol and its derivatives, together with a significant amount of proteinaceous material (~10%). These components may in fact derive from the body itself, rather than being a result of the embalming process. Although phospholipids are susceptible to degradation and as such would not be expected to survive intact, the presence of the amides and proportion of the

fatty acids seen in the aged tissues of Khnumnakht may reflect the presence of phospholipids (Buckley et al. 1999, p.443-452). The mummy of Khnumnakht was also highly polymerised in nature, as indicated by the characteristic alkene/alkane doublets seen in the Py-GC/MS analysis.

The two XVIIth dynasty mummies (5.4.3 & 5.4.4) were dominated by free fatty acids, produced as a result of the hydrolysis of triacylglycerols which would have been present in the original fat/oil. The samples which were in direct contact with the body from both the adult and child were very similar, the C_{16:0} to C_{18:0} ratios (1.5 and 1.2) and cholesterol derivatives present probably indicating their derivation from the body itself. In contrast, the stained wrapping from the child and the textile/fatty matter from the adult were notably different from those of the tissues, and remarkably similar to each other in saturated fatty acid composition, which is particularly notable given that they do not indicate a human origin. The C_{16:0} to C_{18:0} ratios were 0.9 for both mummies, the presence of cholesterol markers clearly indicative of an animal origin. Furthermore, these ratios along with the branched chain fatty acids would strongly indicate a ruminant origin, most probably sheep fat since this has a notably higher C_{18:0} abundance compared to the C_{16:0} fatty acid, with a ratio of 0.9 for mutton fat (Mills & White 1994, p.33) in comparison to an average of 1.2 for beef (Mills & White 1994, p.33). The use of sheep fat in these Theban mummies would not be unexpected, given the association of the ram with the local Theban god Amun, suggesting the potential desire to be associated with the deity and imbued with his qualities. The high abundance of oleic acid and low amounts of oxidised fatty acids, in all but the stained wrapping, suggests that these samples are remarkably well preserved.

The two samples from the New Kingdom head (5.4.5) were very similar, and dominated by fatty acids with a C_{16:0} to C_{18:0} ratio (3.4) indicative of a plant oil. Minor amounts of a coniferous resin were also confirmed by the presence of 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid detected by GC/MS. This was supplemented by the TD-GC/MS analysis which revealed the thermolytically derived methyl dehydroabietate, methylation of the free diterpenoid acids taking place in the probe at the 310°C temperature used for TD.

In both the TD and solvent extracts, the major constituents of the Third Intermediate Period female adult mummy (5.4.6) were fatty acids, including appreciable quantities of α,ω -dicarboxylic acids (C₄ to C₁₄) with C₈ and C₉ predominating. The high ratio of C_{16:0} to

C_{18:0} (4.4) suggests that is a highly oxidised plant oil, accounting for ~60% of the extractable material. The components identified in both the TD analysis and solvent extracts included a significant abundance of the diterpenoids 7-oxodehydroabietic and 15-hydroxy-7-oxo-dehydroabietic acids, and the thermolytically-derived methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate, in addition to a number of decarboxylated 7-oxo-diterpenoids. Together with retene and 15-dehydroretene, these confirmed the presence of a coniferous pitch (~6%) which had undergone strong heating before being applied to the body. Further major components included a series of *n*-alkanes, C₂₅ to C₃₃, with an odd-over-even preference. This pattern of alkanes with the C₂₇ dominant is indicative of beeswax (any wax esters which often occur with the alkanes would not be expected to elute under the chromatographic conditions and column employed). In the acid fraction long chain fatty acids C_{22:0} to C_{30:0}, maximising at C_{24:0} were present, again indicating a likely beeswax origin since these acids are the main free fatty acids found in beeswax. The presence of beeswax is also strongly supported by the unusual 15-hydroxyhexadecanoic acid, which derives from the hydrolysis of the hydroxy wax esters, and is the major hydroxy acid (72%) obtained on hydrolysis of beeswax (Tulloch 1971, p.235-265). The solvent extracts contained the *n*-alkanes (C₂₁ to C₃₃ maximising at C₂₇) with odd-over-even preference, which were observed in the TD together with wax esters (C₄₀ to C₅₀, maximising at C₄₆). Although no intact hydroxy wax esters were detected, the presence of the alkanes and wax esters, together with the long chain fatty acids and 15-hydroxyhexadecanoic acid, confirms that beeswax makes up about 30% of the solvent soluble extracts. Hydroxyaromatic acids observed as minor components indicated the possible presence of a balsamic resin (1.5%), although storax can be eliminated as a possible source.

In the analysis of two samples taken from the mummy of 'Neskhons' (5.4.7), the major components in both were fatty acids with a C_{16:0} to C_{18:0} ratio of 1.7. The presence of both cholesterol and plant sterols also indicated the use of a mixture of animal fat and plant oil, with the sample locations making it unlikely that these could have derived from the body itself. The appreciable abundance of α,ω -dicarboxylic acids indicated that the fat/oil mixture had undergone a significant degree of oxidation. Present as trace components were 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid, confirming a coniferous resin in both samples, although no pitch markers such as retene were identified. Wax esters in the C₄₀ to C₅₀ range were detected as relatively minor components, along with the C₂₇ and C₂₉ *n*-alkanes (also observed in the TD analysis), although the presence of

the latter compounds as only very minor components, along with the observation of the more dominant C₂₂ and C₂₄ alkenes suggests that hydrocarbons potentially diagnostic of a particular wax were highly degraded in this sample leaving the precise origin and nature of the wax uncertain. Triterpenoid acids were also present in both samples, and included a prevalence of dammaranes (e.g. shoreic type acids) indicative of a dammar resin (from *Diterocarpaceae*, indigenous to south-east Asia) (van der Doelen et al. 1998, p.249-264), present in significant quantities (4%) in the wrappings taken from both the neck and hip areas. A highly degraded *Pistacia* resin can have similar chemistry to a degraded dammar, however, the absence of moronic acid (often used to distinguish between two resins) and norolean-17-en-3-one in the TD/Py and solvent extracts, a major component in strongly heated *Pistacia* (Columbini et al. 2000, p.19-29), would suggest that dammar resin was a significant component of the embalming 'resin'. Unfortunately the abundances of the triterpenoid acids were too low to obtain good mass spectra which might aid a more positive identification. A sugar/gum was tentatively identified as a significant constituent (~5%) in both wrappings – not unreasonable given the ancient Egyptian's supposed use of plant gums (e.g. acacia) to secure the linen wrappings as part of the embalming procedure. The hydroxyaromatic acids again observed as minor components indicated the possible presence of a balsamic resin (~1%), although once again storax may be ruled out.

The three samples analysed from the mummy of 'Pedeamun' (664 to 404 BC) (5.4.8) were very similar in qualitative composition. Fatty acids dominated all samples, the 'resin' from the top of the head for example containing about 55% fat/oil and the C_{16:0} to C_{18:0} ratio of 5.0 confirming its origin as a plant oil. Present in the samples were saturated long-chain acids in the C_{22:0} and C_{30:0} carbon number range with lignoceric predominant, together with a series of *n*-alkanes (C₂₅ to C₃₃) maximising at C₂₇ with an odd-over-even preference and wax esters (C₄₀ to C₅₀ maximising at C₄₆) consisting predominantly of the C_{16:0} acyl group. Hydroxy wax esters (C₄₄ to C₅₀) were also present, albeit as minor components, thereby confirming the presence of ~40% beeswax (see Figure 5.12). A coniferous resin was also identified, evidenced by the detection of trace quantities of the dehydroabietic and 7-oxodehydroabietic acids, with minor amounts of hydroxyaromatic acids indicative of a balsamic resin/umbelliferae, although storax can once more be excluded.

Notably, the Py-GC-MS profiles for all three samples taken from the mummy of Pedeamun are remarkably similar, all of them dominated by a series of alkene/alkane doublets in the carbon number range C₉ to C₂₃ (see Fig. 5.13b) with longer chain 1-alkenes

also dominant (C_{24} to C_{32}) in samples [1] and [5]. These latter compounds clearly derive from the wax esters, the alkenes containing the same number of carbon atoms as the long chain alcohol constituting the respective wax ester. Thus, C_{30} would be expected to be the major long chain alkene, deriving from the cleavage of the O-CH₂ ester bond with cis-elimination in the C_{46} wax ester to give palmitic acid and the C_{30} 1-alkene, i.e. with the carbon number corresponding to alcohol chain length in the original ester. The relative abundances of the long chain alkenes (C_{24} to C_{32}) do indeed reflect the distribution of the wax esters, the large amount of $C_{16:0}$ fatty acid in the pyrogram also reflecting the release of this palmitic acid on cleavage of the O-CH₂ bonds in the wax esters. The alkene/alkane doublets C_9 to C_{23} maximising at C_{11} are also present in all the samples in this study which contain beeswax, thus providing a very distinctive fingerprint. Figure 5.20 provides a comparison of the pyrolysis profiles of fresh and aged beeswax. The pyrogram of fresh beeswax does not display any of the *n*-alkanes observed in the archaeological samples (which are presumed to reflect the greater degree of saturation typical of aged, degraded beeswax). However, the relative distributions of the *n*-alkenes (C_8 - C_{23} and C_{24} - C_{32}) along with the $C_{16:0}$ fatty acid released from the wax esters are remarkably similar. This is of major significance, since sequential TD-GC/MS and Py-GC/MS clearly has the potential to positively identify beeswax without the need for solvent extraction procedures, whilst requiring the very small size of sample so attractive to museum curators and archaeologists.

The two samples from the female Ptolemaic adult (5.4.9) were remarkably similar, despite having originated from opposite ends of the body. Both contained significant amounts of the alkanes, wax esters, hydroxy wax esters and methyl 15-hydroxyhexadecanoate confirming the presence of beeswax, with triterpenoid acids, including isomasticadienonic, masticadienonic, oleanonic and the major triterpenoid moronic acid characteristic of a *Pistacia* resin, the presence of polyfunctionalised triterpenoids confirming its oxidised nature. Minor amounts of hydroxy aromatic acids were also found, indicating the presence of a balsamic resin or *Umbelliferae*. Perhaps most notable however, were the later eluting wax esters (C_{48} to C_{60} , and maximising at C_{52} , with the $C_{24:0}$, $C_{26:0}$ and $C_{28:0}$ acyl groups dominant) which appear to have no alkanes associated with them. These wax esters do not originate from the beeswax in the sample (see Fig. 5.21a), furthermore, the presence of only C_{48} to C_{60} wax esters with the $C_{26:0}$ acyl group predominant is characteristic of Chinese insect wax (Fig. 5.21b; Tulloch 1973, p.367-371), a commodity which was clearly

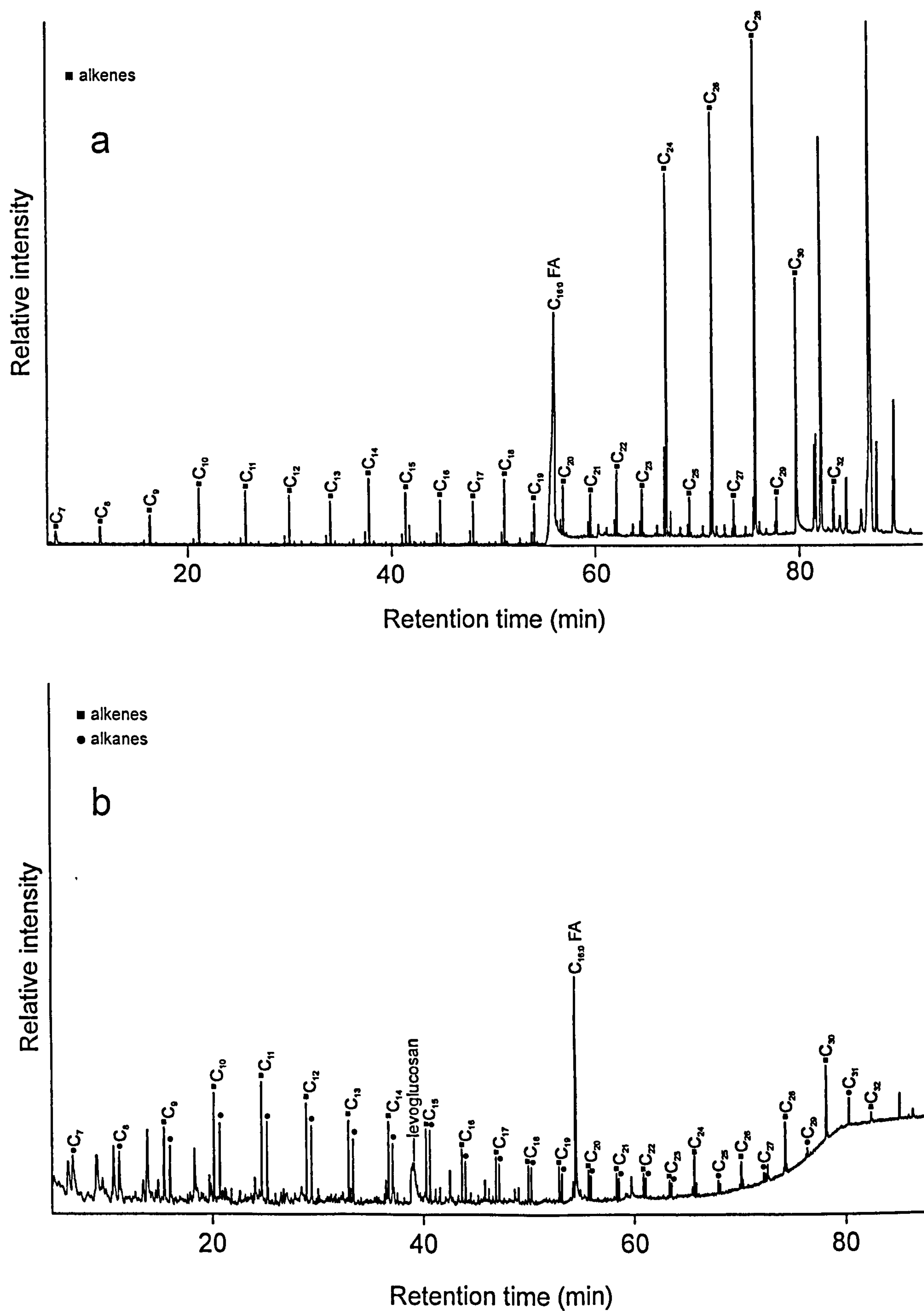


Figure 5.20 Total ion chromatograms of the pyrolysis profiles (610°C/10s, after TD at 310°C/10s) of (a) fresh beeswax and (b) 'resin' from the upper edge of the cartonnage opposite the right cheek/zygomatic bone [5] of the Third Intermediate Period male adult 'Pedeamun', XXVIth-XXVIIth dynasty (c. 664-404 B.C.).

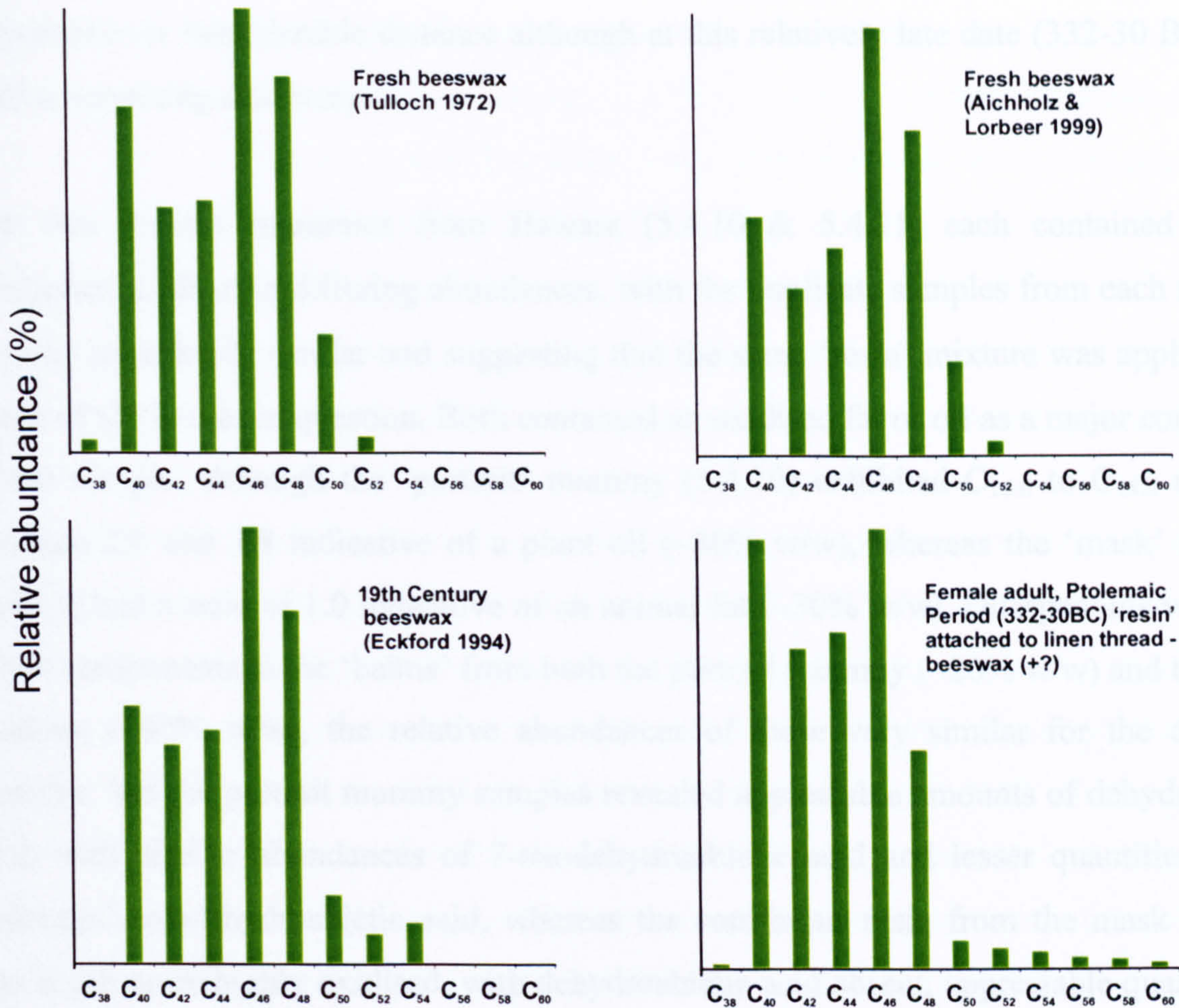


Figure 5.20a Histograms showing the distributions of wax esters of even carbon number in fresh beeswax and archaeological samples containing beeswax

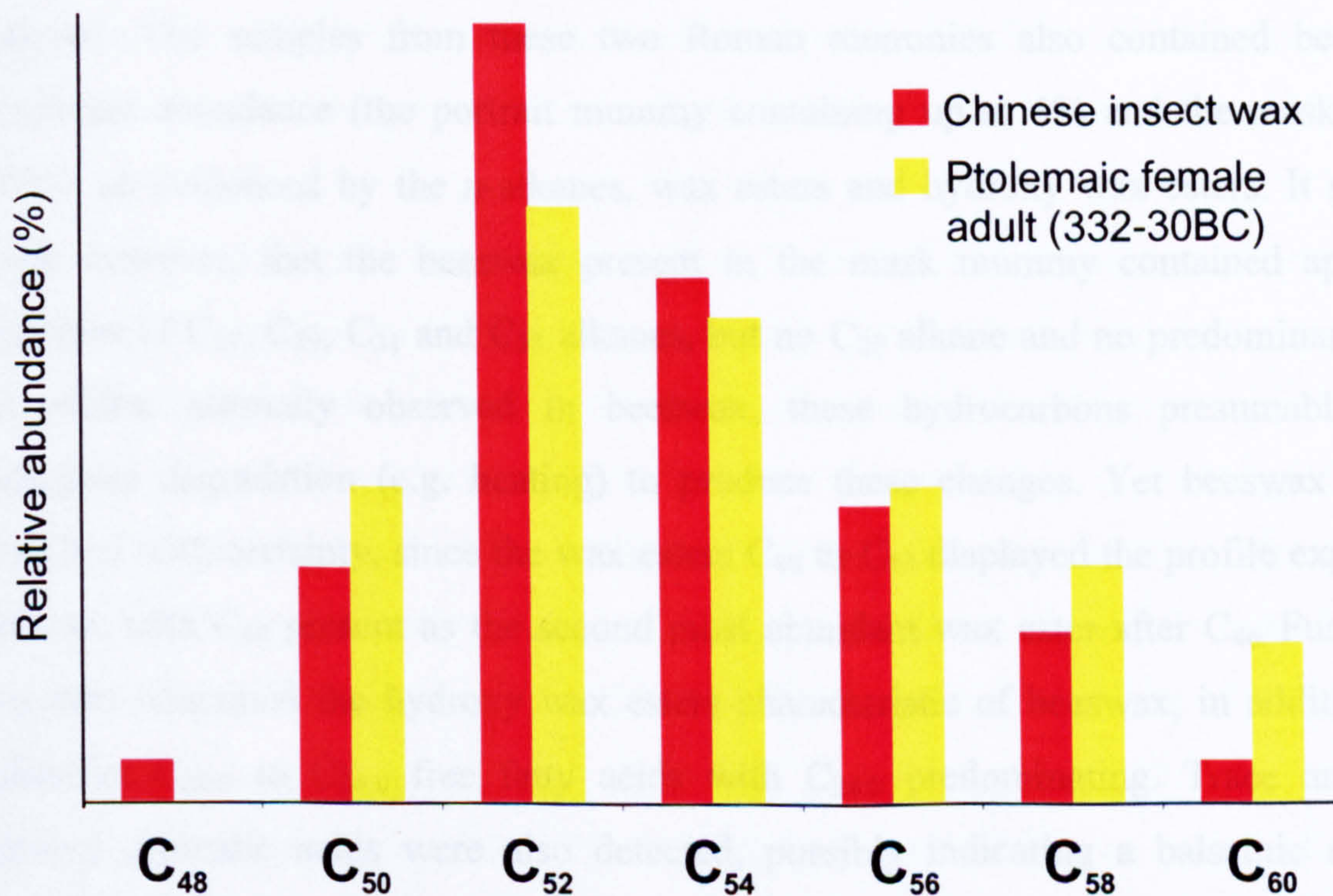


Figure 5.21b. Histogram showing a comparison of the wax ester distributions (C₄₈ to C₆₀, even carbon numbers) of Chinese insect wax and the 'resin' attached to linen thread from the right ankle of the Ptolemaic female adult (c. 332-30 B.C.).

The abundance of the C₄₈ wax ester (containing the C_{24:0}, C_{26:0} and C_{28:0} long chain acyl groups likely to derive from Chinese insect wax) in the 'resin' from the Ptolemaic female adult could not be determined due to the predominance of the C₄₈ palmitate originating from the beeswax present.

The abundance of the 'Chinese insect wax' derived C₅₀ wax ester present in the 'resin' from the Ptolemaic female adult is an approximation based on the intensity of long chain acyl groups (C_{24:0}, C_{26:0} and C_{28:0}) in its mass spectrum.

imported over considerable distance although at this relatively late date (332-30 B.C.) not such a surprising discovery.

The two Roman mummies from Hawara (5.4.10 & 5.4.11) each contained similar components, albeit in differing abundances, with the duplicate samples from each mummy proving remarkably similar and suggesting that the same 'resin' mixture was applied over much of the bodies in question. Both contained an oxidised fat or oil as a major constituent of the sample, although the 'portrait' mummy (5.4.10) exhibited $C_{16:0}$ to $C_{18:0}$ ratios of between 2.0 and 3.8 indicative of a plant oil (~80% w/w), whereas the 'mask' mummy (5.4.11) had a ratio of 1.0 indicative of an animal fat (~30% w/w). Diterpenoids were also major components in the 'balms' from both the portrait mummy (~20% w/w) and the mask mummy (~40% w/w), the relative abundances of these very similar for the duplicate samples. Yet the portrait mummy samples revealed appreciable amounts of dehydroabietic acid, with similar abundances of 7-oxodehydroabietic acid and lesser quantities of 15-hydroxy-7-oxodehydroabietic acid, whereas the coniferous resin from the mask mummy was much more highly oxidised, with dehydroabietic acid absent, appreciable quantities of 7-oxodehydroabietic acid and very considerable quantities of 15-hydroxy-7-oxodehydroabietic acid, this later diterpenoid being the main component of both samples analysed. The samples from these two Roman mummies also contained beeswax in significant abundance (the portrait mummy containing up to 6% and the mask mummy ~30%), as evidenced by the *n*-alkanes, wax esters and hydroxy wax esters. It should be noted, however, that the beeswax present in the mask mummy contained appreciable quantities of C_{27} , C_{29} , C_{31} and C_{33} alkanes, but no C_{25} alkane and no predominance of the C_{27} alkane normally observed in beeswax, these hydrocarbons presumably having undergone degradation (e.g. heating) to produce these changes. Yet beeswax could be identified with certainty, since the wax esters C_{40} to C_{50} displayed the profile expected for beeswax with C_{40} present as the second most abundant wax ester after C_{46} . Furthermore, they also contained the hydroxy wax esters characteristic of beeswax, in addition to the indicative $C_{22:0}$ to $C_{34:0}$ free fatty acids with $C_{24:0}$ predominating. Trace amounts of hydroxy aromatic acids were also detected, possibly indicating a balsamic resin; this included a cinnamic acid which could potentially derive from storax.

The last of the two mummies studied were the two Roman male children – the so-called 'twins' (5.4.12 & 5.4.13). As in the case of their adult counterparts (5.4.10 & 5.4.11), both the 'wrapped' and 'unwrapped' twin contained appreciable amounts of a coniferous resin

(8 to 23 & 13% respectively). The unwrapped twin also contained the hydroxy aromatic acids seen in the Roman adult mummies (5.4.10 & 5.4.11), possibly originating from a balsamic resin (excluding storax). The main components, however, were fatty acids which included usually high abundances of $C_{12:0}$, $C_{14:0}$, $C_{16:1}$ and $C_{18:1}$ fatty acids. Despite the relatively unusual fatty acid profile of the 'balms' used in these so-called twins, a positive identification has not yet been achieved. Although the samples taken from the wrapped mummy were relatively degraded compared to those taken from the unwrapped mummy, the relatively high abundances of certain fatty acids present ($C_{12:0}$, $C_{14:0}$, $C_{20:0}$, $C_{22:0}$ and $C_{24:0}$) along with the diterpenoid acids, served to confirm that the same mixture was indeed applied to both mummies, both externally and internally. This is an important fact given the questions surrounding the relationship of the two infants, one of whom died at 9 months old, the other at about 18 months. Most notable however, was the detection of isopimaric acid, albeit as a minor component, this pimarane type diterpenoid only surviving in favourable environments. Its survival would suggest that it was a major diterpenoid acid in the fresh resin, and if so one of the most likely sources would be the Aleppo pine (*Pinus halepensis*) which contains 39% isopimaric (of the total diterpenoids) and is both the major diterpenoid and the most dominant pimarane acid present. Unfortunately, the suggestion must at this stage remain tentative. The presence of isopimaric acid (along with the abundant monounsaturated fatty acids) suggests that the sample is relatively undegraded, as does the abundance of the monounsaturated fatty acids.

A summary of the results of this study is given in Table 5.2. The major products seen in all mummies are degraded acyl lipids, most probably derived from animal fats or plant oils. Where samples were taken directly from the bodies they may well derive from endogenous body lipids, e.g. triacylglycerols from fats or cell membrane phospholipids, and the presence of cholesterol would be consistent with this. However, in cases where degraded fats or oils are seen in the wrappings for example, and not directly in contact with the body, they most likely derive from their intentional application as part of the embalming process. In the majority of cases (i.e. in 7 of the 13 examples, Fig. 5.22 f-l), the fatty acid distributions suggest plant origins, i.e. the high abundance of $C_{16:0}$ as compared with $C_{18:0}$. Beyond this, the identification of the precise plant origin based on fatty acids alone is not possible, due to degradation of major unsaturated components and evidenced by the presence of oxidations products, namely di- (C_7 - C_9) and hydroxycarboxylic acids (C_{18} with the hydroxyl group at C-8, C-9, C-10 and C-11, indicative of autoxidation). Likewise, sterols (cholesterols and phytosterols) are absent in most cases due to their known

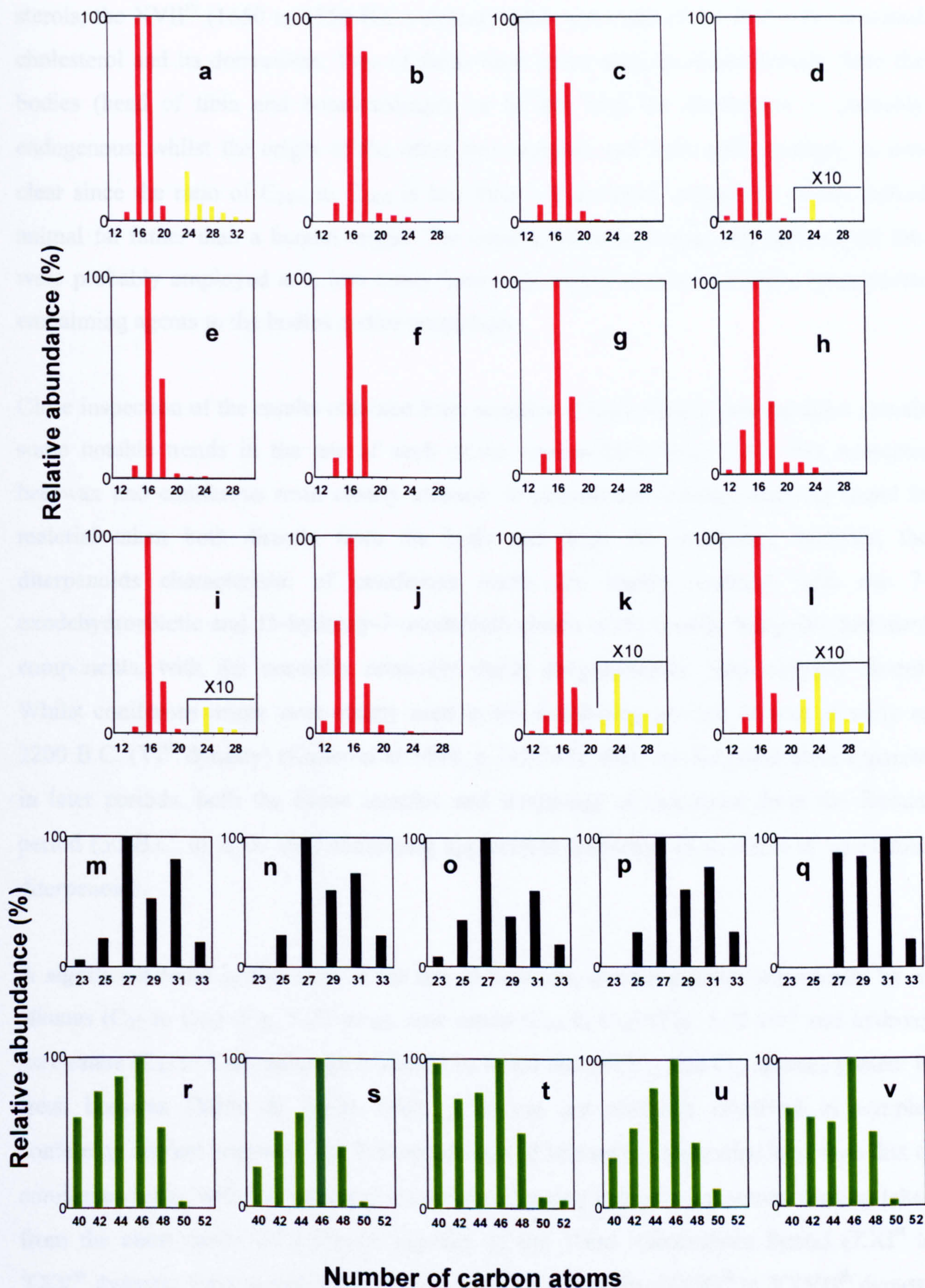


Figure 5.22 Histograms showing the distributions of: (i) fatty acids of even carbon number (fat/oil derived in red, beeswax derived in yellow) in acid fractions from samples taken from the human mummies [see Table 5.2] (a to l); (ii) hydrocarbons of odd carbon number in neutral fractions from samples taken from mummies containing beeswax (m to q); (iii) wax esters of even carbon number in neutral fractions from samples taken from mummies containing beeswax (r to v).

susceptibility to oxidation (Smith 1996, p.453-469). Of the mummies that did display sterols, the XVIIth (1650 to 1550 B.C.) dynasty adult and child (5.4.3 & 5.4.4) contained cholesterol and its derivatives. Two of these were tissue samples taken directly from the bodies (head of tibia and bone/cartilage), so in this case the cholesterol is probably endogenous, whilst the origin of the other two samples, one from each mummy, is less clear since the ratio of C_{16:0} to C_{18:0} is less than 1.0 and more suggestive of an applied animal fat rather than a human origin. The conclusion is that plant oils and animal fats were probably employed as a less costly base with which to mix and apply more exotic embalming agents to the bodies and/or wrappings.

Close inspection of the results obtained from samples of such widely varying dates reveals some notable trends in the use of such exotic commodities (Fig. 5.23). For example, beeswax and coniferous resin clearly increase in prominence through time, as found in material taken both directly from the body and from the wrappings. Notably, the diterpenoids characteristic of coniferous resins are highly oxidised with the 7-oxodehydroabietic and 15-hydroxy-7-oxodehydroabietic acids usually being the dominant components, with the normally relatively stable dehydroabietic acid virtually absent. Whilst coniferous resins were clearly used in the embalming process at least as early as 2200 B.C. (VIth dynasty) (Koller et al 1998, p.343-344), their use becomes most apparent in later periods, both the tissue samples and wrappings of mummies from the Roman period (30 B.C. to A.D. 395) containing appreciable quantities (8 to 44%) of coniferous diterpenoids.

A significant trend is also seen in the use of beeswax, as characterised chemically by *n*-alkanes (C₂₅ to C₃₃) (Fig. 5.22 m-q), wax esters (C₄₀ to C₅₀) (Fig. 5.22 r-v) and hydroxy wax esters (C₄₂ to C₅₄), although it should be noted that the C₃₁ and C₃₃ alkenes present in fresh beeswax (Mills & White 1994, p.50) are not normally observed in samples containing ancient beeswax. The first appearance of beeswax is somewhat later than that of coniferous resin, with its earliest positive identification being in a resinous coating taken from the chest cavity of a female mummy of the Third Intermediate Period (XXIst to XXVth dynasty; 1069 to 664 B.C.). In a sample taken from the XXVIth to XXVIIth dynasty (664 to 404 B.C.) mummy Pedeamun, the solvent soluble extracts (constituting 89%) comprise 43% beeswax (Fig. 5.12), which falls within the range of 25 to 50% found in over half of the beeswax containing 'balms'. An even greater amount was found in two samples of 'resin' from a female mummy of Ptolemaic date (332 to 30 B.C.) (Fig. 5.14), in

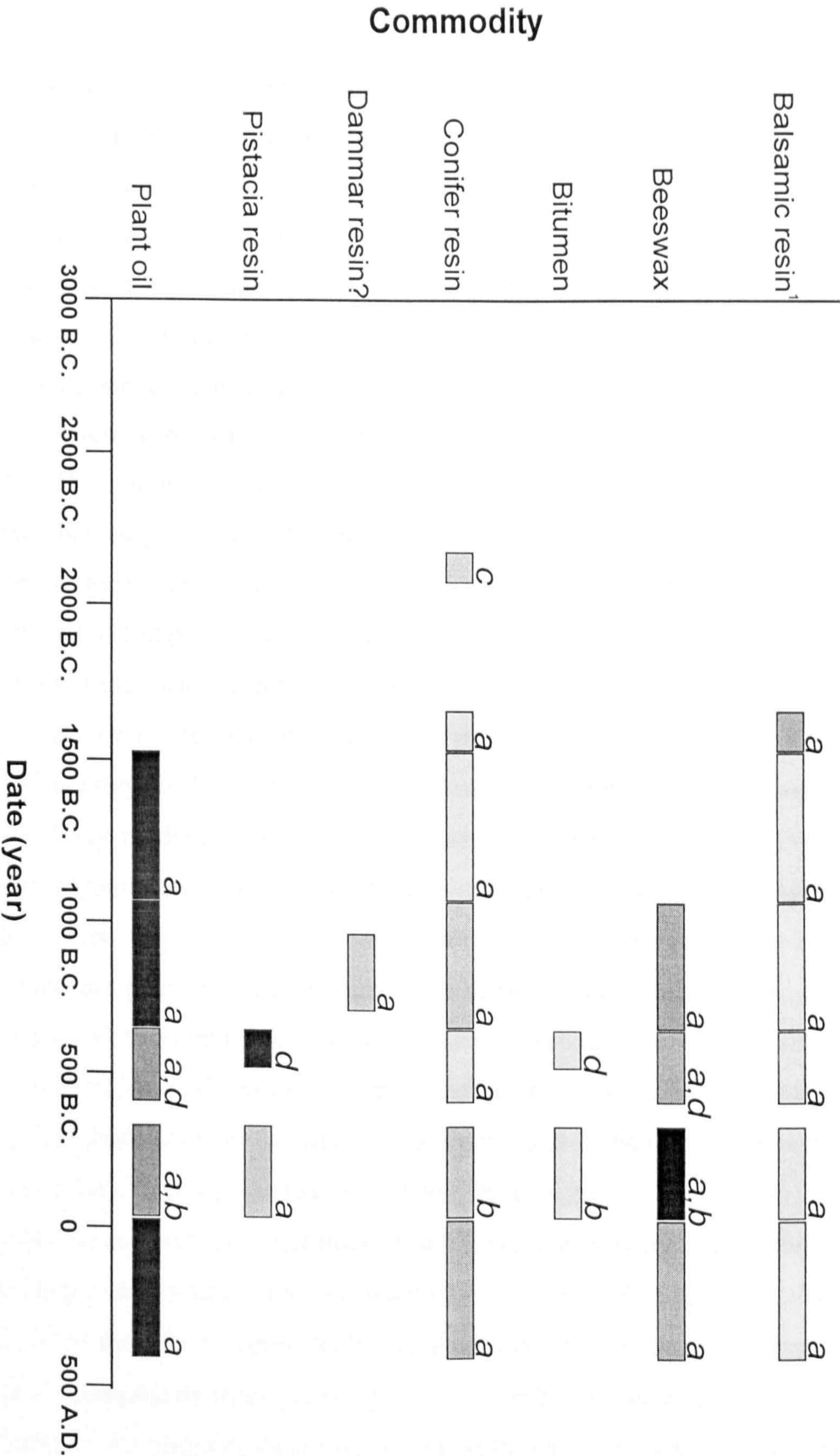


Figure 5.23. The use of organic commodities (preservatives/unguents) identified in chemical investigations of the 'balms' of provenanced and dated ancient Egyptian mummies. Grey scale reflects therelative abundance of a particular commodity found in the balm samples, with darker shades representing a higher relative abundance. ¹ tentative identification of balsamic resins. ^a data from this study; ^b Rullkotter & Nissenbaum, 1988; ^c Koller *et al.*, ^d Colombini *et al.* 2000.

which beeswax made up 85 to 89% of the solvent extractable components, i.e. this treatment was probably beeswax with the addition of a small amount of *Pistacia* resin, oil/fat and Chinese insect wax.

Other components of the embalming 'resin' are triterpenoids and hydroxyaromatic acids diagnostic of plant products. Comprising the former are isomasticdienonic, masticdienonic, moronic and oleanonic acids diagnostic of the presence of *Pistacia* resin (Mills & White 1994, p.107-108), as found in the Ptolemaic female mummy (Fig. 5.14) where the *Pistacia* content constitutes 5% of the extractable lipids. Triterpenoid acids, including a prevalence of dammaranes (e.g. shoreic type acids) indicative of a dammar resin (from *Dipterocarpaceae*, indigenous to south-east Asia) (van der Doelen et al 1998, p.249-264) were also present in significant quantities (4%) in the wrappings of the XXIInd dynasty (945 to 715 B.C.) mummy Neskons. Although a highly degraded *Pistacia* resin can have a similar chemistry to degraded dammar, the absence of moronic acid (often used to distinguish between the two resins) and norolean-17-en-3-one in the TD/Py and solvent extracts (a major component in strongly heated *Pistacia*, Colombini et al 2000, p.19-29), would suggest that dammar resin was a significant component of the embalming 'resin' of this mummy (van der Doelen et al 1998, p.249-264). Yet, given the use of dammar in the 19th century, in both oil painting and museum artefact conservation, the possibility of a relatively modern source must be considered. Radiocarbon dating of the triterpenoids, using preparative GC prior to AMS dating, would provide the answer to this important question. Less easy to assign an origin for are the hydroxyaromatic acids, although clearly these are plant derived. Present in 10 of the 13 mummies studied, their distribution is generally dominated by the 4-hydroxy-3-methoxybenzoic acid, with smaller amounts of p-hydroxybenzoic acid consistent with oxidation of unsaturated C-3 side chains (either acid or alcohol substituted) in the aromatic benzoate ester of balsamic resins, or of ferulic acid present as a major component in *Umbelliferae* (Serpico 2000, p.450). The absence of the precursor compounds is not unexpected, given the susceptibility of these to the oxidation prevalent in mummies. Although sawdust was often used to pack the cavities of mummies, their derivation from lignin can be ruled out due to the absence of the major lignin building-block compounds (methyl, ethyl, *n*-propyl and vinyl guaiacols) in any of the Py-GC/MS analyses. An origin of these hydroxy aromatic acids in storax (*Liquidamber orientalis*) can in all but one case be ruled out, based on the absence of cinnamic acids and triterpenoids. These are dominant in storax (Pastorova et al 1998, p.1381-1393), the

methoxy aromatic derivatives found being absent in storax but major components of other balsamic resins (Pastorova et al 1998, p.1381-1393; Lucas 1989, p.95).

These results reveal the use of a mixture of commodities, with a compositional diversity greater than has been previously reported. Despite the frequent use of the term 'bitumen' in connection with mummification, the idea that its general use in embalming has now been proven "unequivocally" (Bahn 1992, p.109) cannot be supported. In repeat searches the steranes and hopanes characteristic of petroleum bitumens (Rullkotter & Nissenbaum 1988, p.618-621) were not detected, leading to the conclusion that natural bitumens were not used in these mummies. Furthermore, given that beeswax generally constitutes the greater proportion of the organic materials rather than the resins, the term embalming 'wax' has at least equal validity to so-called 'resin'. In the crass assumption that Egyptian mummification is now fully understood, these results clearly illustrate that the materials used by the ancient Egyptian embalmers were far more diverse than previously documented.

5.6 CONCLUSIONS

The characterisation and identification of organic materials employed in the mummification of 14 human mummies (including 'Horemknesi'; Chapter 3) has been carried out using GC, GC/MS and sequential TD-GC/MS and Py-GC/MS. This investigation has resulted in a number of significant conclusions.

- (i) Sequential TD-GC/MS and Py-GC/MS have shown their value in aiding the identification of a wide range of organic materials including fats/oils, proteins, sugar/gum, coniferous resin, *Pistacia* resin and beeswax. For a fairly wide range of materials TD gave a useful 'fingerprint', comparable with the more conventional solvent extracts. However, polyfunctional components were not observed, and given the likely abundance of such polar molecules in aged samples this is a notable limitation.
- (ii) The use of beeswax, certainly where it is present as a significant constituent, can be positively identified by sequential TD-GC/MS and Py-GC/MS, thus providing a virtually non-destructive method of analysis for the characterisation and identification of this important commodity.

- (iii) Bitumen was not present in these samples, as demonstrated by the absence of any bitumen markers in any of the samples analysed, despite searches using mass chromatograms for the steranes (m/z 217) and hopanes (m/z 191) characteristic of natural bitumens.
- (iv) The detection for the first time of a dammar resin in a XXIInd dynasty (945-715 B.C.) mummy, as demonstrated by the prevalence of dammaranes and an absence of moronic acid which is characteristic of *Pistacia*. It should be said, however, that further work (specifically dating) is necessary in order to exclude the possibility of a modern origin for this resin.
- (v) The use of Chinese insect wax for the first time in an ancient Egyptian mummy, as demonstrated by the presence of C₄₈ to C₆₀ wax esters, maximising at C₅₂, together with the C_{24:0} C_{26:0} C_{28:0} free fatty acids which form the dominant acyl groups in the wax esters.
- (vi) The use of beeswax at least as extensively as resins, as demonstrated by the presence of C₂₅ to C₃₃ alkanes, together with C₄₀ to C₅₀ wax esters and C₄₂ to C₅₄ hydroxy wax esters.

CHAPTER 6

Animal mummies: an historical comparison

CHAPTER 6: ANIMAL MUMMIES – AN HISTORICAL COMPARISON

6.1 OBJECTIVES

This chapter involves the chemical investigation of animal mummies in an attempt to characterise and identify the organic materials employed in their embalming. Samples of both wrappings and ‘resin’ were taken from provenanced and dated mummies, with the investigation focussing on examples from the ‘classic’ mummy-making era of pharaonic/dynastic Egypt in an attempt to understand something of the development of animal mummification, an area which has been very much neglected to date. Animal mummification remains a contentious area of ancient Egyptian culture, the production of literally millions of such mummies generally interpreted to suggest that relatively little care and expense was involved in their preparation in comparison with human mummies. Yet the ancient Egyptians generally treated animals with great respect, regarding them as representatives of the gods (e.g. the cat represented the goddess Bastet; the hawk, Horus; the ibis, Thoth and so forth). A comparison between human and animal mummies, with anatomical considerations taken into account, might therefore provide significant insights into important facets of ancient Egyptian culture. GC, GC/MS and sequential TD-GC/MS and Py-GC/MS were utilised to facilitate the characterisation and identification of the organic embalming agents employed over some six hundred years of large-scale animal mummy production.

6.2 INTRODUCTION

Prior to this thesis there had been no rigorous studies carried out on provenanced and dated animal mummies to determine the organic embalming agents employed. Using the methods of chemical analysis (GC/MS, etc.) capable of identifying such complex degraded residues, the following study examined samples from 4 provenanced and dated animal mummies (two hawks, a cat and an ibis). All four have been dated to the pharaonic/dynastic period and were selected as examples of ‘classic’ Egyptian embalming as opposed to later Graeco-Roman examples. Although the number of animals studied is modest, it nevertheless marks an important starting point, with factors such as animal type, cultic influences and body location taken into account wherever possible.

The animal mummies included in this study are shown in Table 6.1, together with the samples analysed. A multiple sampling approach (as demonstrated by Serpico and

Table 6.1. Provenance and date of animal mummies studied and the origin of samples analysed.

Mummy	Date/age	Provenance	Sample location and description ^c
Hawk ^a ^b 52.55.46	818-664 BC (XXIII rd -XXV th dynasty)	Tarkhan	'Resin' on wrapping underneath jaw/mandible [2] 'Resin' on back, base of neck (head of spine) [3] 'Resin' on wrapping covering right breast [4]
Hawk ^a ^b 52.55.47	818-664 BC (XXIII rd -XXV th dynasty)	Tarkhan	'Resin'-soaked linen above right eye [2]
Cat ^a ^b 56.22.224	664-332 BC (XXVI th -XXX th dynasty)	Beni Hassan	Blackened wrapping from base of mummy [1] Detached 'resin'-soaked wrapping 1 [2] Detached 'resin'-soaked wrapping 2 [3] Red material in right ear [5]
Ibis ^a ^b 1969.112.42	664-332 BC (XXVI th -XXX th dynasty)	Sakkara	'Resin'-soaked wrapping, covering right breast [1] 'Resin'-soaked wrapping, covering left breast [2]

^a Liverpool Museum; ^b museum number; ^c the term 'resin' denotes physical appearance and does not presuppose any chemical composition or biological origin.

White 1998, p.1037-1048) was again employed for the animal mummies, and although samples were taken from as many different locations on the body as possible, the relatively good state of preservation of the mummies allowed only the wrappings and resin-like material to be analysed. Obviously the greater number of samples examined, the more accurate and meaningful the picture obtained, and although time constraints restricted the analysis of all the samples available; those chosen hopefully provided the most meaningful information within these limits.

Given the sensitive nature of the material concerned, the sampling of animal mummies must necessarily be pragmatic and it therefore concentrated on areas of the mummies already accessible/exposed and/or damaged. Yet care was taken to avoid potentially contaminated areas already exposed, and sample sizes taken reflected the need to obviate any problems of this nature, in addition to difficulties associated with highly oxidised/degraded material on the surface of the resins, waxes, fats, etc.

Diagnostic marker compounds present in the original embalming agents and resistant to degradation can be related to specific embalming agents and were therefore used to

identify the 'balms'. GC/MS and TD/Py-GC/MS were used to facilitate the molecular separation and identification of these marker compounds. Due to the nature and history of the proposed embalming materials, both free and polymerised components were likely to be present (see 2.5). Therefore the 'dual' approach of GC/MS (following solvent-extraction procedures) and sequential TD-GC/MS and Py-GC/MS was employed to allow the characterisation and identification of both the free (solvent-extractable) marker compounds and the recognisable sub-units of polymeric materials not amenable to the more conventional GC/MS approach.

6.3 BACKGROUND

The potential origin of the organic embalming agents has been discussed above (Chapters 1 & 2), together with the archaeological and historical background of these materials. In summary, true resins (conifer and Pistacia), gum (e.g. frankincense and myrrh) and balsamic (e.g. storax) resins, beeswax, bitumen, animal fats, plant oils and others (Tables 1.3, 2.1) are all possible sources of embalming materials. Yet the indiscriminate use of the terms 'resin' and 'bitumen' perpetuate the extreme ignorance surrounding the nature of the organic materials used in mummification, a situation the current study seeks to begin to rectify. It has also been argued (e.g. Lucas, 1989, p.304; Malek, 1997, p.123-134) that animal mummification was relatively inexpensive, with little regard for the quality and effectiveness of the embalming undertaken. It is further suggested that the organic materials employed would have been the cheaper commodities available, such as oils/fats and bitumen (Lucas 1989, p.304), presuming a desire to avoid expense in the mummification of animals. Yet given the respect the ancient Egyptians afforded such creatures, and the fact that many were symbolic of their deities, this would seem a rash and misguided assumption.

The diverse chemistry of the organic embalming materials has also been described above (Chapter 2, 2.3) and so will not be detailed here. The chemistry of each of the samples analysed will be dealt with in turn in the results section and related back to their likely origin. In general, degradation processes such as dehydration, aromatisation, polymerisation and particularly oxidation are likely to have taken place over the 2,300 to 2,800 years the samples remained *in situ*. It is important to reiterate previous comments (see Chapter 5), i.e. that only two of the four provenanced and dated studies carried out outside Bristol mention the identification of highly oxidised components in the organic

materials they analysed, the first identifying oxidised diterpenoids (Koller et al. 1998, p.343-344) and the second mentioning oxidised triterpenoids, although their exact nature was not discussed (Colombini et al. 2000, p.19-29). Given the samples' likely treatment (e.g. heating, etc.) and their likely post-depositional environment, this is a significant omission, since it is likely that oxidised constituents will be present in appreciable abundance (Serpico & White 1998, p.1037-1048; Proefke et al. 1992, p.105.A-111.A; Weser et al. 1998, p.511.A-516.A; Buckley et al. 1999, p.443-452). Interpretations must be made with circumspection, given that mixtures may well have been used (Herodotus II, 83-90, 1954, p.160-161; Diodorus in Smith & Dawson 1924, p.62-63), and an open mind is a crucial prerequisite if the picture obtained is to be at all meaningful.

6.4 RESULTS

The focus of this study was the characterisation and identification of the organic embalming agents employed in each of the animal mummies investigated. As before, a further important aspect of the research was to assess the value of sequential TD/GC-MS and Py-GC/MS which is of particular value given the small sample sizes required (~0.1 mg) and the limited sample preparation involved. A comparison of the data obtained from each technique for a given sample and mummy will be presented in order to allow a direct comparison of the data obtained by each technique, particularly a comparison between TD-GC/MS and the more conventional GC/MS. The subsequent discussion will then, where appropriate, make general comparisons of the findings and the relative merits of the techniques utilised, together with the implications of the information they have provided.

For a summary of the findings of this study see Table 6.2. The identification of the compounds observed was based on both their mass spectra (NIST/EPA/NIH Mass Spectral Database) and retention times. For TD/Py-GC/MS the compounds are present as the free compounds. The compounds identified in the solvent extracts (total lipid extract, acid and neutral fractions) are present as the free compounds or as their TMS derivatives.

Table 6.2. Provenance and date of animal mummies, origin of ‘balms’ and their composition.

Mummy	Date/age	Provenance	Sample location and description ³	Inferred components of embalming “resin” ⁴	Relative abundance (%) ⁵
Hawk ¹ ² 52.55.46	818-664 BC (XXIII rd -XXV th dynasty)	Tarkhan	‘Resin’ on wrapping underneath jaw/ mandible [2]	Fat/oil Wax (beeswax?) ^{a,f}	90% 10%
			‘Resin’ on back, base of neck (head of spine) [3]	Fat/oil Wax (beeswax?)	90% 10%
			‘Resin’ on wrapping covering right breast [4]	Fat/oil Wax (beeswax?)	90% 10%
Hawk ¹ ² 52.55.47	818-664 BC (XXIII rd -XXV th dynasty)	Tarkhan	‘Resin’-soaked linen above right eye [2]	Fat/oil Wax ^{b,g}	30% 70%
Cat ¹ ² 56.22.224	664-343 BC (XXVI th – XXX th dynasty)	Beni Hassan	Blackened wrapping from base of mummy [1]	Animal fat Cedar resin Pistacia resin Balsam/umbelliferae ⁴ Beeswax ^{c,h} Gum resin - myrrh? Cinnamon?	60% 4% 4% 1% 30% 1% trace?
			Detached ‘resin’-soaked wrapping 1 [2]	Animal fat Cedar resin Balsam/umbelliferae ⁴ Beeswax ^{d,i} Gum resin - myrrh?	61% 5% 1.5% 31% 1.5%
			Detached ‘resin’-soaked wrapping 2 [3]	Animal fat Cedar resin Pistacia resin Balsam/umbelliferae ⁴ Beeswax ^{e,j} Gum resin - myrrh?	64% 2% 0.2% 0.5% 33% 0.5%
			Red material in right ear [5]	A sugar/gum Plant oil Beeswax	95% 5% 0.5%
			‘Resin’-soaked wrapping, covering right breast [1]	A sugar/gum Plant oil Wax	100% trace trace
Ibis ¹ ² 1969.112.42	664-343 BC (XXVI th -XXX th dynasty)	Sakkara			

Ibis (contd)				'Resin'-soaked wrapping, covering left breast [2]	A sugar/gum Plant oil Wax	100% trace trace
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¹ Liverpool Museum ² museum number; ³ the term 'resin' denotes physical appearance and does not presuppose any chemical composition or biological origin; ⁴ of the 3 cases where components indicative of a degraded balsam were found, none could originate from the 'local' balsamic resin storax (*Liquidambar orientalis*); ⁵ % relative abundance based on absolute concentrations, calculated based on internal standards added at the extraction stage (TD taken into account where appropriate). Superscript letters (a-j) refer to the histograms shown in Fig. 6.13.

6.4.1 Hawk, XXIIIrd –XXVth dynasty (c.818-664 BC), Third Intermediate/Late Period, Tarkhan (52.55.46)

6.4.1.1 'Resin' on wrapping underneath lower jaw/mandible [2]

GC/MS – Total lipid extract

The results for the total lipid extract analysed by GC/MS are shown in Figure 6.1a. A range of carboxylic acids was identified, including the monocarboxylic acids (C₁₄ to C₂₈) with C_{16:0} and C_{18:0} being the main two components in decreasing order of abundance. Yet these often dominant components of many of the samples analysed were only in relatively moderate abundance in comparison to the *threo* isomer of 9,10-dihydroxyoctadecanoic acid which was the major constituent of the extract. This later compound was characterised by *m/z* 73, 129, 147, 215 and 317, the *erythro* isomer present only as a relatively minor component eluting just after the *threo* isomer. The C_{16:1} and C_{18:1} unsaturated acids were present as minor constituents of the extract, as were saturated long-chain fatty acids in the C_{22:0} and C_{28:0} carbon number range, with C_{24:0} predominating. Interestingly, the α,ω -dicarboxylic (C₆ to C₁₁) and monohydroxycarboxylic acids, usually seen in oxidised fats or oils (Gulaçar et al. 1989, p.61-72; Gulaçar et al. 1990, p.691-705; Regert et al. 1998, p.2027-2032), were not present. Observed as minor constituents, were a series of *n*-alkanes (C₂₅ to C₃₁ maximising at C₂₇) with an odd-over-even preference, along with wax esters in the C₄₂ to C₅₀ carbon number range, with C₄₆ predominating. The C₄₂ and C₄₄ wax esters contained predominantly the C_{16:0} acyl group (*m/z* 257) with a moderate abundance of C_{18:0} (*m/z* 285), the C₄₆ ester consisted predominantly of the C_{18:0} acyl group, but with a significant proportion of C_{16:0} and the C₄₈ ester of largely the C_{18:0} acyl group with smaller amounts of C_{20:0} and C_{22:0}. The C₅₀ wax ester was in too low a concentration to obtain any meaningful information on the nature of the acyl group(s) present. Of similar abundance to the *n*-alkanes were the C₂₄, C₂₆ and C₂₈ *n*-alkanols with a more significant amount of the 1-C_{18:0} monoacylglycerol. Also present in significant abundance in the chromatogram was a phthalate, a common plasticiser and contaminant.

6.4.1.2 'Resin' on back, base of neck (head of spine) [3].

GC/MS – Total lipid extract

The results for the total lipid extract analysed by GC/MS are shown in Figure 6.1b. Monocarboxylic acids (C₁₄ to C₁₈) were identified with C_{16:0} and C_{18:0} being the main two components in decreasing order of abundance. Yet again, the palmitic and stearic acids were less abundant than the *threo* isomer of 9,10-dihydroxyoctadecanoic acid which was a

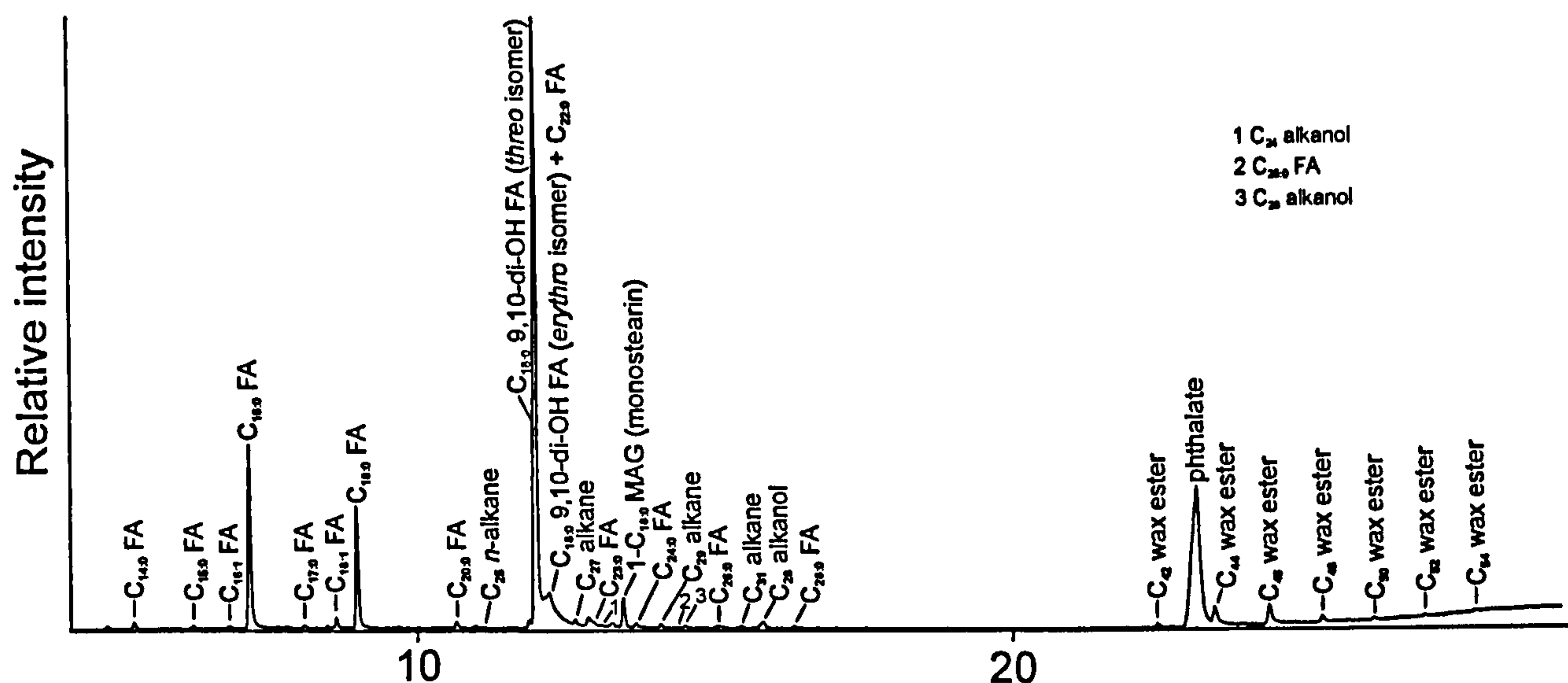


Figure 6.1a Total ion chromatogram from the GC/MS analysis of the total lipid extract of 'resin' on the wrapping underneath the lower jaw/mandible [2] of the Third Intermediate/Late Period hawk '52.55.46', XXIIIrd-XXVth dynasty (c. 818-664 B.C.).

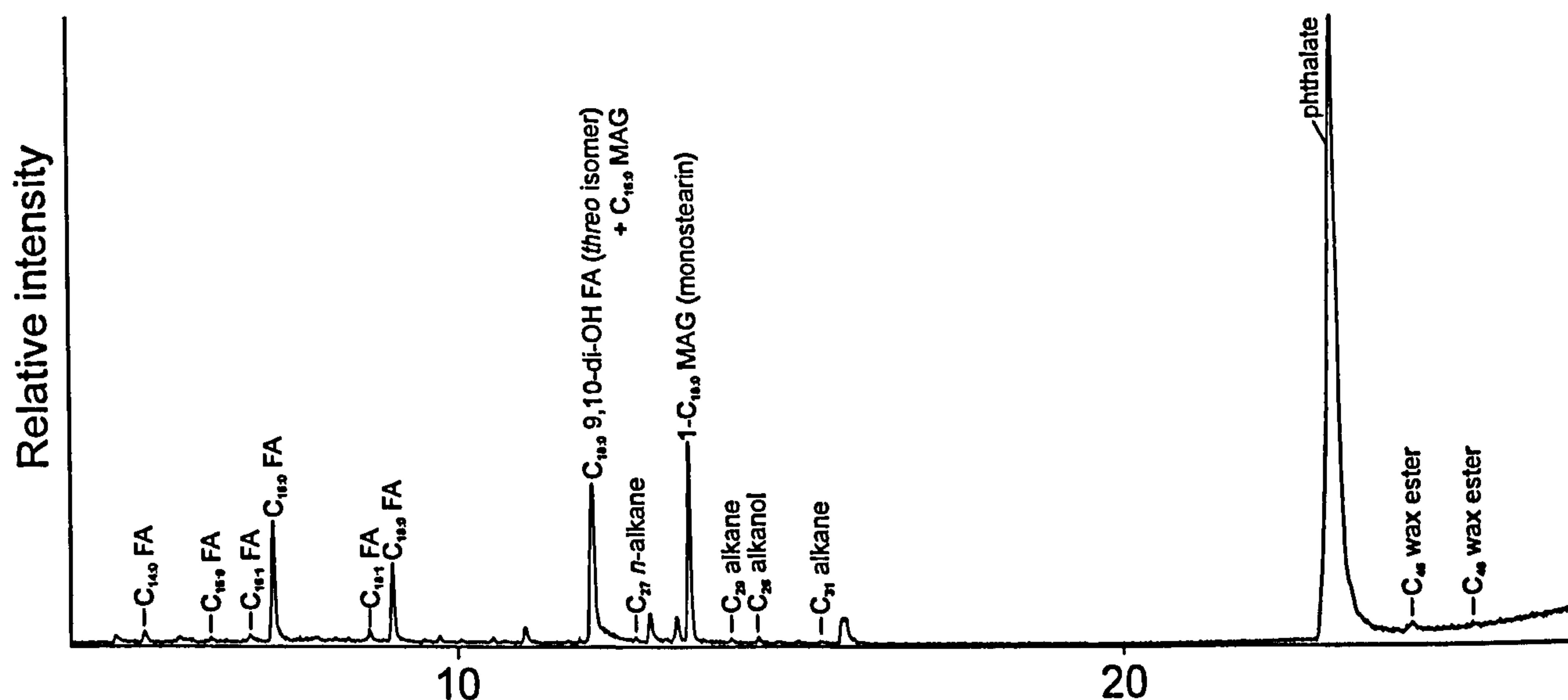


Figure 6.1b Total ion chromatogram from the GC/MS analysis of the total lipid extract of 'resin' on the back, at the base of the neck (head of spine) [3] of the Third Intermediate/Late Period hawk '52.55.46', XXIIIrd-XXVth dynasty (c. 818-664 B.C.).

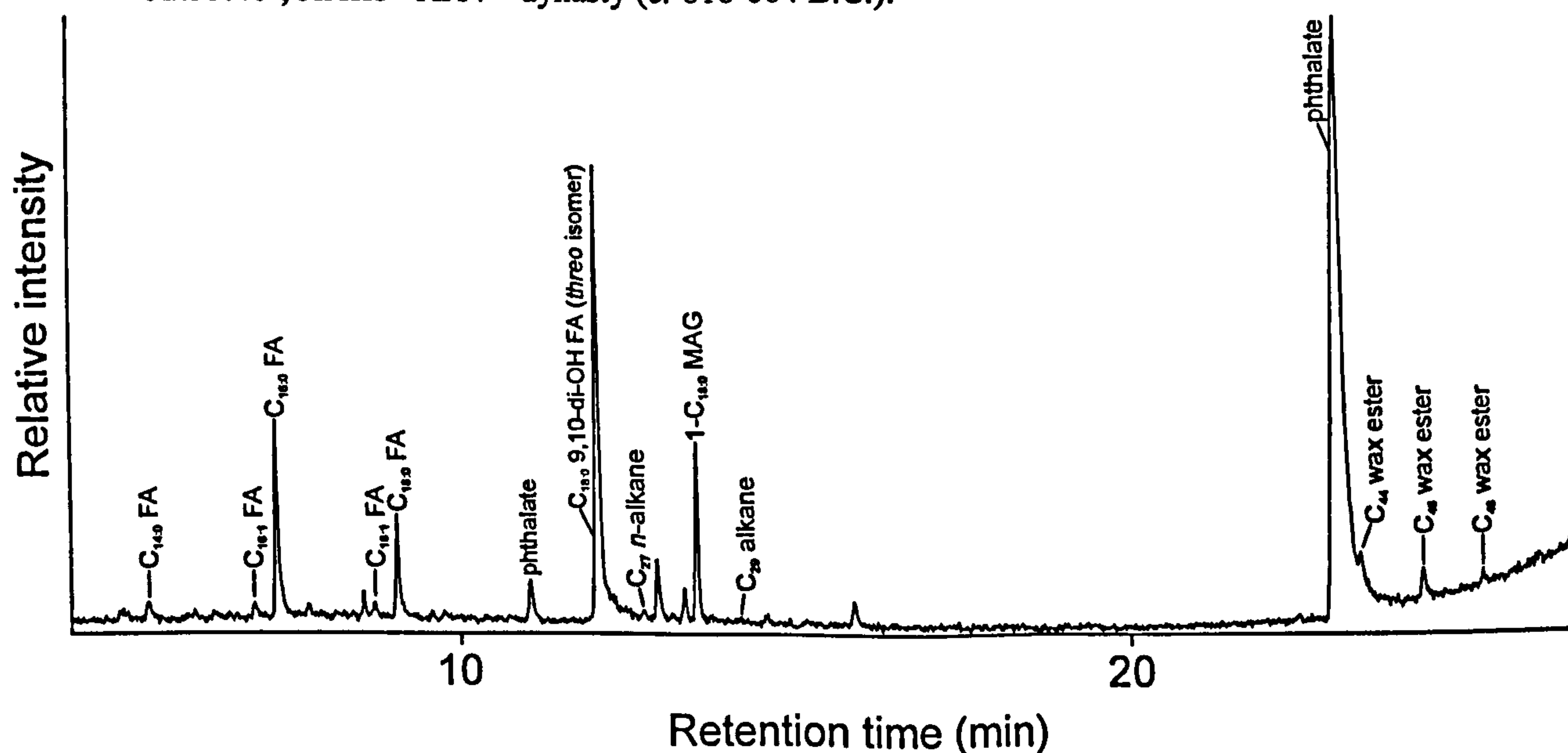


Figure 6.1c Total ion chromatogram from the GC/MS analysis of the total lipid extract of 'resin' on wrapping covering the right breast [4] of the Third Intermediate/Late Period hawk '52.55.46', XXIIIrd-XXVth dynasty (c. 818-664 B.C.).

major constituent (as with sample 6.4.1.1, the *erythro* isomer was present only as a relatively minor component) of the extract along with the 1-C_{18:0} monoacylglycerol. The 1-C_{16:0} monoacylglycerol was also a significant component. The C_{16:1} and C_{18:1} unsaturated acids were present as trace constituents of the sample and again, the α,ω -dicarboxylic (C₆ to C₁₁) and monohydroxycarboxylic acids, usually seen in oxidised fats or oils, were absent. Also observed as trace constituents, were the C₂₇ to C₃₁ *n*-alkanes, maximising at C₂₇ with an odd-over-even preference, together with the C₄₆ and C₄₈ wax esters, with C₄₆ predominating. Of similar abundance to the *n*-alkanes was the C₂₆ *n*-alkanol. The major component in the chromatogram was in fact a phthalate, as mentioned above a common plasticiser and contaminant.

6.4.1.3 'Resin' on wrapping covering right breast [4].

GC/MS – Total lipid extract

The results for the total lipid extract analysed by GC/MS are shown in Figure 6.1c. Monocarboxylic acids (C₁₄ to C₁₈) were identified with C_{16:0} and C_{18:0} being the main two components in decreasing order of abundance. Yet again the palmitic and stearic acids were less abundant than the *threo* isomer of 9,10-dihydroxyoctadecanoic acid which was the major constituent (as with samples 6.4.1.1 and 6.4.1.2, the *erythro* isomer was present only as a relatively minor component) of the extract. The 1-C_{18:0} monoacylglycerol was also a major component with a lesser amount of the 1-C_{16:0} monoacylglycerol. The C_{16:1} and C_{18:1} unsaturated acids were present as trace constituents of the sample and yet again, the α,ω -dicarboxylic (C₆ to C₁₁) and monohydroxycarboxylic acids, usually seen in oxidised fats or oils, were absent. Also observed as trace constituents, were the C₂₇ and C₂₉ *n*-alkanes, with C₂₇ predominating, together with the C₄₄, C₄₆ and C₄₈ wax esters, maximising at C₄₆. The major peak in the chromatogram was again the phthalate contaminant.

6.4.2 Hawk, XXIIIrd –XXVth dynasty (c.818-664 BC), Tarkhan (52.55.47)

6.4.2.1 'Resin'-soaked linen above right eye/orbit [2]

GC/MS – Total lipid extract

The results for the total lipid extract analysed by GC/MS are shown in Figure 6.2. Monocarboxylic acids (C₁₆ to C₃₂) were identified with C_{16:0} and C_{18:0} being the main two components in decreasing order of abundance. The C_{18:1} unsaturated acid was present as a minor constituent of the sample, as were saturated long-chain fatty acids in the C_{22:0} and C_{32:0} carbon number range, with C_{32:0} predominating. The *threo* and *erythro* isomers of

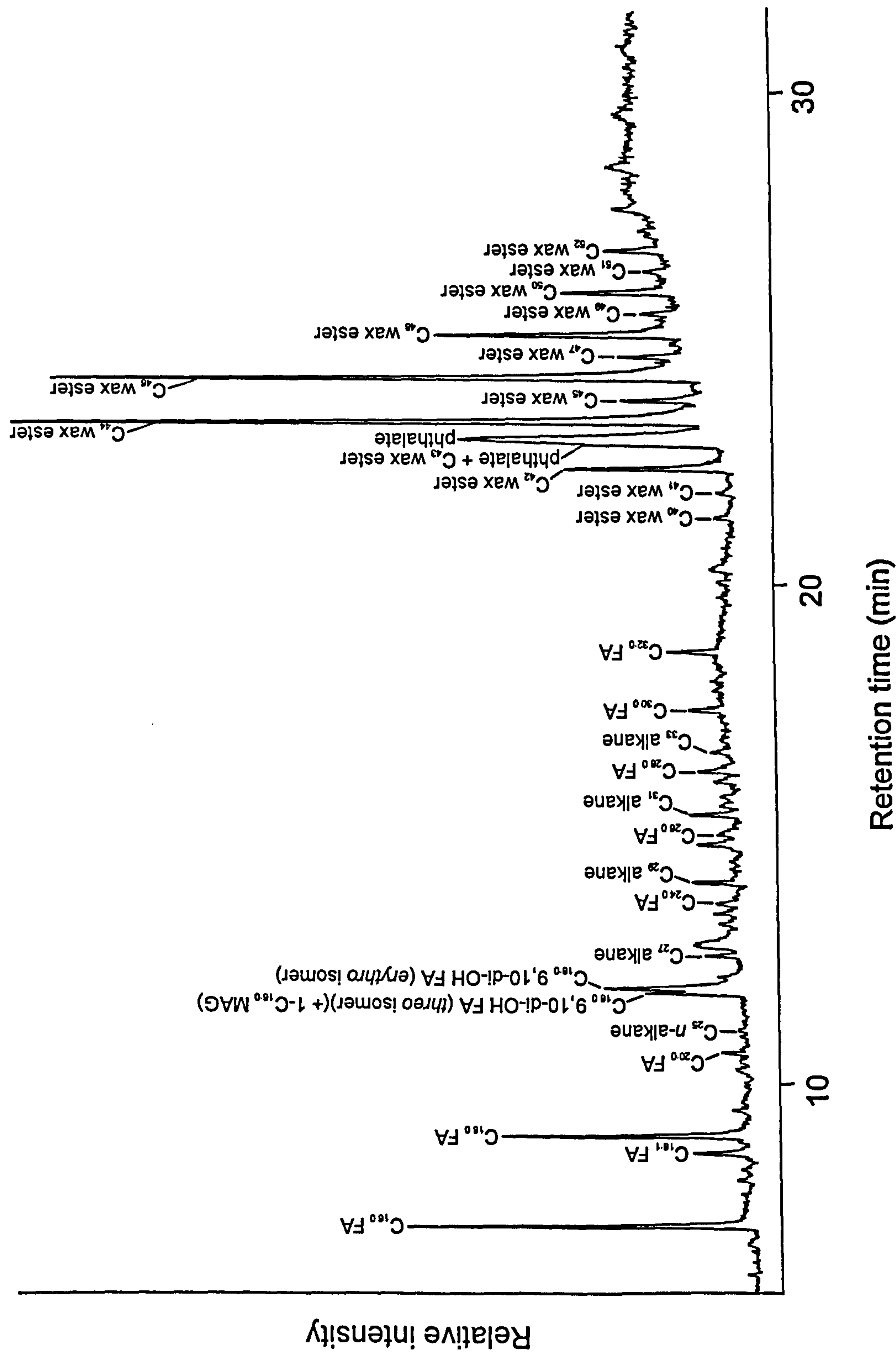


Figure 6.2 Total ion chromatogram from the GC/MS analysis of the total lipid extract of 'resin'-soaked linen above the right eye [2] of the Third Intermediate/Late Period hawk '55.55.47', XXIIIrd-XXVth dynasty (c. 818-664 B.C.)

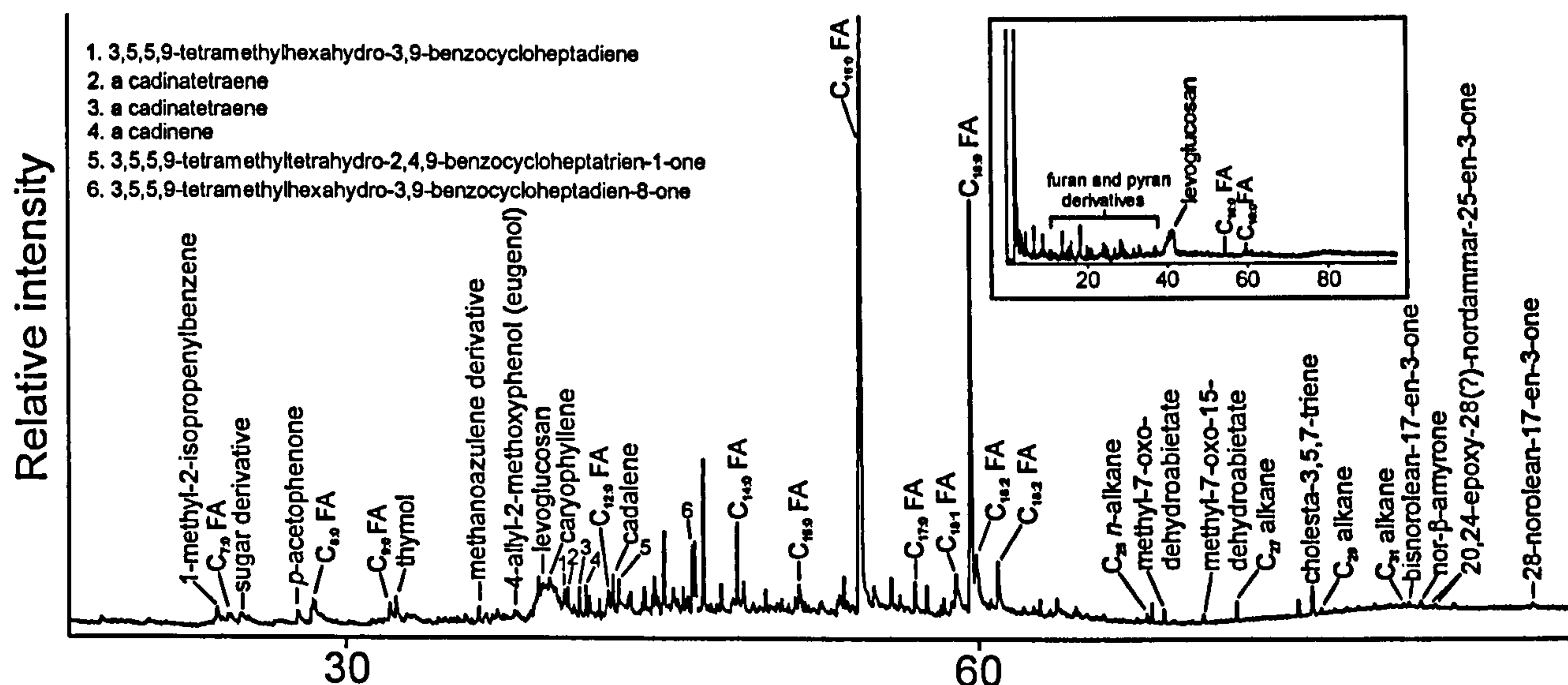
9,10-dihydroxyoctadecanoic acid were significant components of the extract, however, unlike the previous mummified hawk, the isomers were of similar abundance. The α,ω -dicarboxylic (C_6 to C_{11}) and monohydroxycarboxylic acids, usually seen in oxidised fats or oils (refs), were not present. A series of *n*-alkanes (C_{25} to C_{33} maximising at C_{29}) with an odd-over-even preference were observed, along with wax esters in the C_{40} to C_{52} carbon number range, with C_{44} predominating. These wax esters were the major constituents of the total lipid extract. The C_{40} , C_{42} , and C_{44} wax esters contained predominantly the $C_{16:0}$ acyl group (m/z 257), with lesser amounts of $C_{18:0}$ (m/z 285), with the C_{46} and C_{48} wax esters consisting largely the $C_{16:0}$ and $C_{18:0}$ acyl groups, but with lesser proportions of $C_{20:0}$ (m/z 313) and $C_{22:0}$ (m/z 341). The C_{50} component contained predominantly $C_{22:0}$ with significant proportions of the $C_{16:0}$ and $C_{18:0}$ acyl groups (no meaningful information could be obtained on the C_{52} wax ester acyl group(s)). The phthalate observed in the samples from hawk 6.4.1, was also a significant peak in the chromatogram.

6.4.3 Cat, XXVIth–XXXth dynasty (c.664-343 BC), Beni Hassan (56.22.224)

6.4.3.1 Blackened wrapping from base of mummy [1]

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 6.3a. The TD profile displayed a complex pattern with many peaks present. A series of monocarboxylic acids (C_7 to C_{18}) were detected, the major components being $C_{16:0}$ and $C_{18:0}$ in decreasing order of abundance, these last two fatty acids dominating the TD profile. Two $C_{18:2}$ fatty acids were also identified as significant components, with relatively little $C_{18:1}$ and unusually high relative abundances of the short chain fatty acids ($C_{7:0}$ to $C_{9:0}$), suggesting that the original oil or fat had undergone appreciable oxidative change. The steroidal compound, cholesta-3,5,7-triene was detected. A series of *n*-alkanes, C_{25} to C_{31} , with an odd-over-even preference were observed as minor components, as were the diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate. The monoterpene, thymol was present in moderate abundance, along with more appreciable amounts of bicyclic sesquiterpenoids which included a number cadinenes, several substituted benzocycloheptenes and caryophyllene (see Fig. 6.4). The major cadinenes were identified as two cadinatetraenes (M^+ 200), two cadinatrienes (M^+ 202) and the fully aromatised cadalene (M^+ 198). The two oxidised sesquiterpenoids were identified as 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadien-8-one, characterised by m/z 91, 105, 119 (base peak), 161, 203 and M^+ 218 and 3,5,5,9-tetramethyl-tetrahydro-2,4,9-benzocycloheptatrien-1-one, characterised by m/z 91, 117, 132 (base peak) and M^+ 216



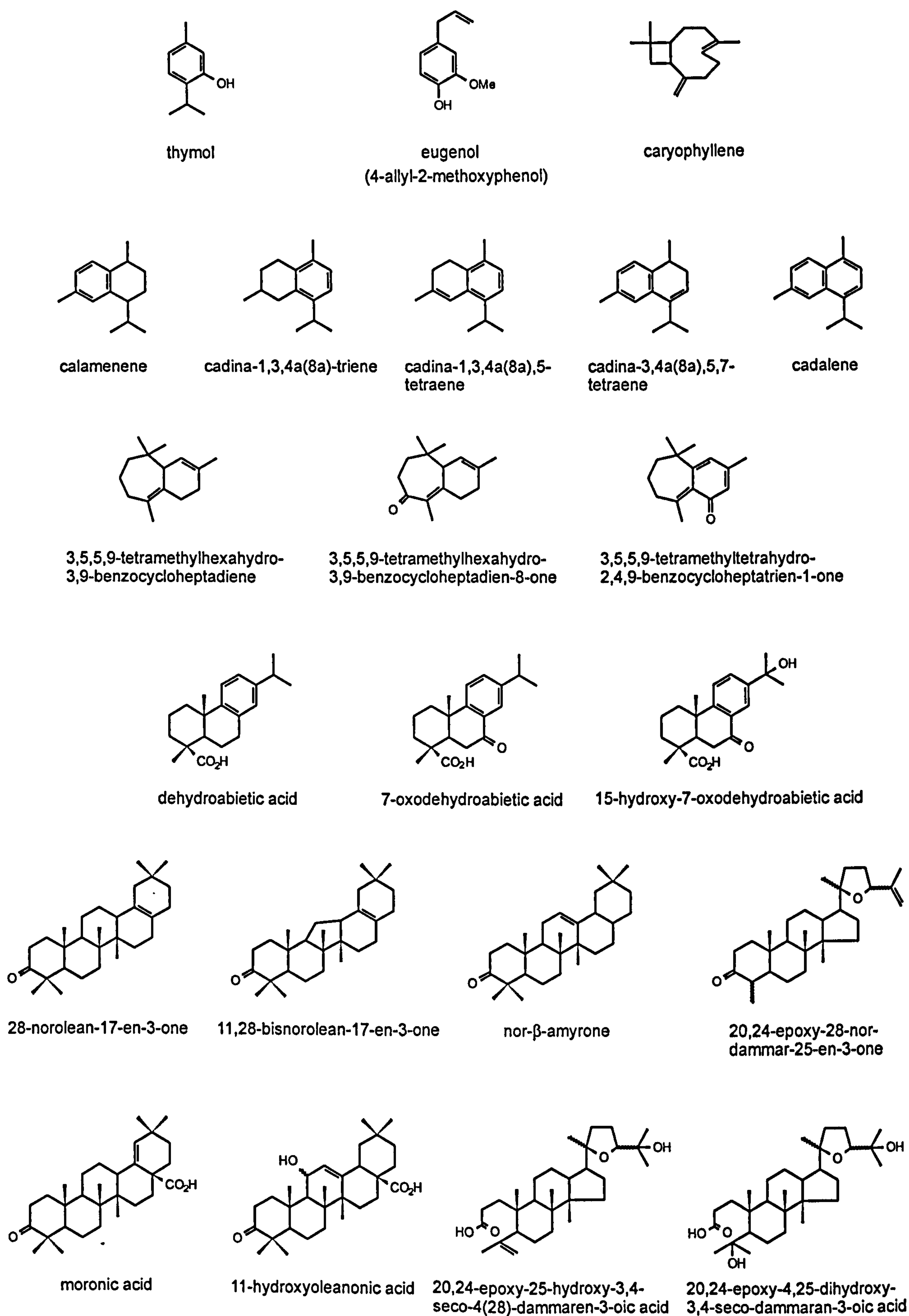


Figure 6.4 The monoterpene, sesquiterpene, diterpene and triterpene compounds identified in the TD-GC/MS and GC/MS analyses of the blackened wrapping from the base of the mummy [1] of the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

(see Fig. 6.5). These two compounds were accompanied by the naturally occurring (see discussion) 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadiene characterised by m/z 91, 105, 119 (base peak), 135, 161, 176 and M^+ 204. The mass fragmentation of the major peaks of these three compounds, and the reaction mechanism for the formation of the 8-one is presented in Figure 6.6. Caryophyllene was also identified by its mass spectrum, characterised by 41 (base peak), 69, 79, 93, 105, 119, 133, 161, 189 and M^+ 204, as was 4-allyl-2-methoxyphenol (eugenol), m/z 77, 91, 103, 149 and M^+ 164 (base peak). The observation of levoglucosan and furan derivatives suggests a possible sugar/plant gum origin, and this is supported by the presence of cadinenes, however, the sugar markers may derive from hydrolysed cellulose present in the degraded linen wrappings (see discussion). As shown in the two XVIIth dynasty mummies, intact cellulose does not produce levoglucosan at the TD desorption temperatures used in this study, cleavage of the cellulose to yield levoglucosan only taking place at the 610°C. At later retention times (76.5 to 87.5 mins) a number of triterpenoids were observed (see Fig. 6.4), the major components being 28-norolean-17-en-3-one (M^+ 410 and base peak m/z 163), nor- β -amyrone [m/z 189 and 204 (base peak)] and bis-norolean-17-en-3-one (M^+ 396 and base peak m/z 163). A number of these triterpenoids were octotillone-type molecules (base peak m/z 143) and their dehydrated analogues (base peak m/z 125), the major of these tentatively identified as 20,24-epoxy-28(?)-nordammar-25-en-3-one (two isomers).

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 6.3a, inset. The $C_{16:0}$ and $C_{18:0}$ fatty acids, most likely deriving from the bound fraction of the sample. A series of cadalenes was also observed (though they were somewhat obscured by the very prominent levoglucosan peak). The main component however, with the exception of CO_2 , was levoglucosan, with appreciable amounts of furan and pyran derivatives, although these almost certainly derive from the cellulose which constitutes the linen wrappings.

GC/MS – Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 6.3b. The major components were monocarboxylic acids (C_{12} to C_{30}), with $C_{16:0}$ and $C_{18:0}$ predominating. The $C_{18:1}$ unsaturated acid was present as a minor constituent of the sample, as were saturated long-chain fatty acids in the $C_{22:0}$ and $C_{30:0}$ carbon number range with $C_{24:0}$ predominating. Significant quantities of α,ω -dicarboxylic acids (C_6 to C_{11}), with C_8 and C_9

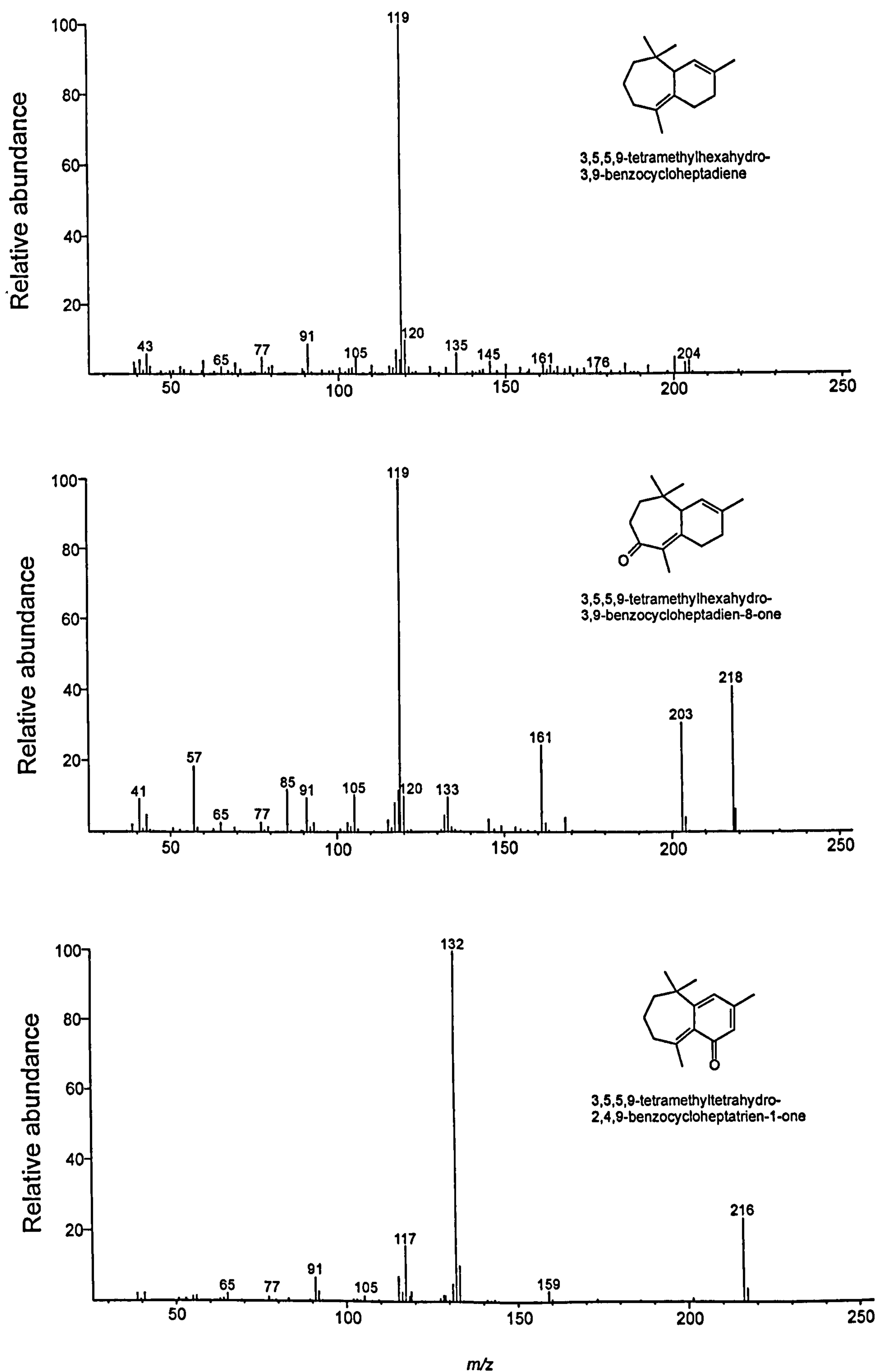


Figure 6.5 The mass spectra of the benzocycloheptenes identified in the TD-GC/MS and GC/MS analyses of the blackened wrapping from the base of the mummy [1] of the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

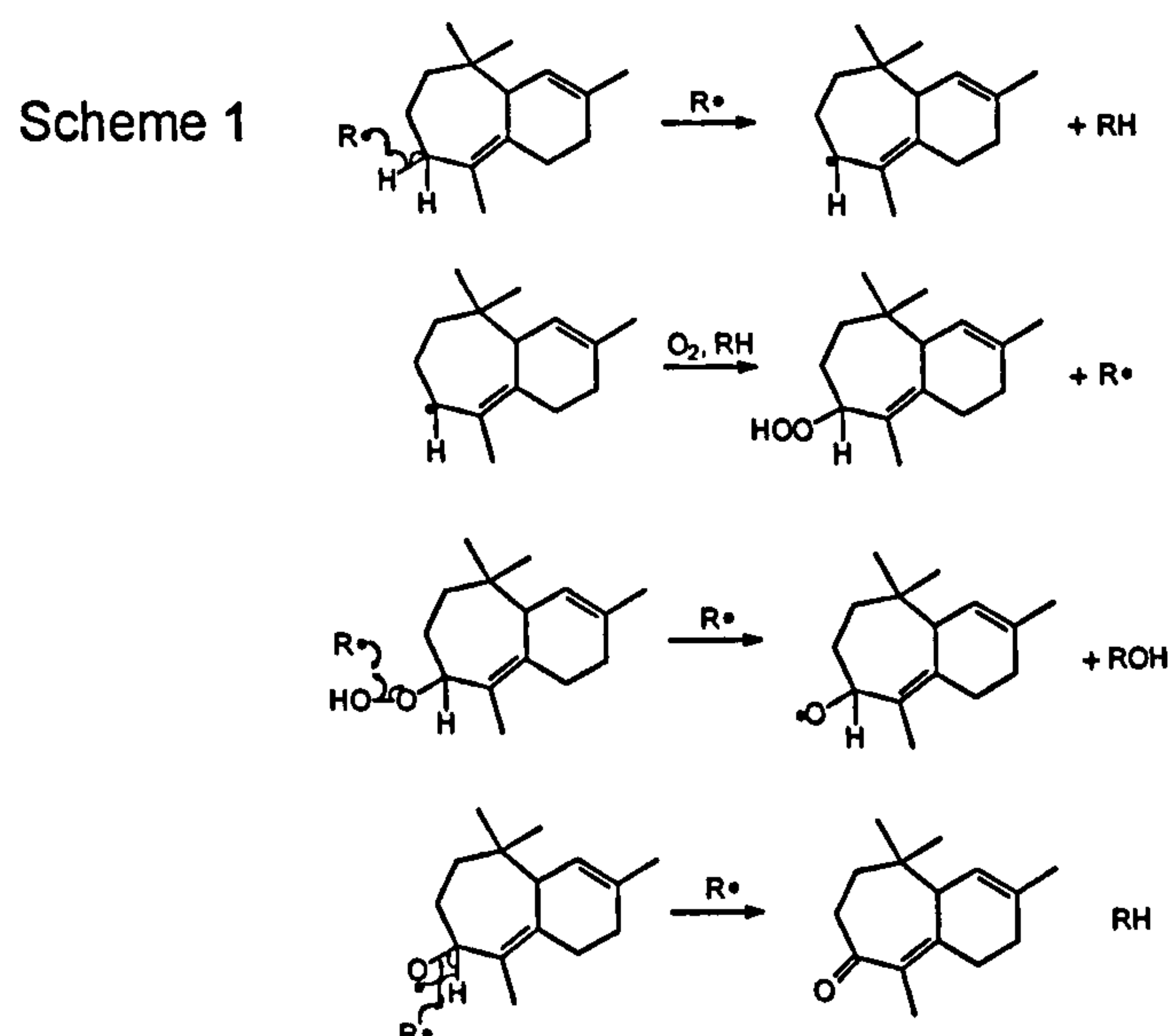
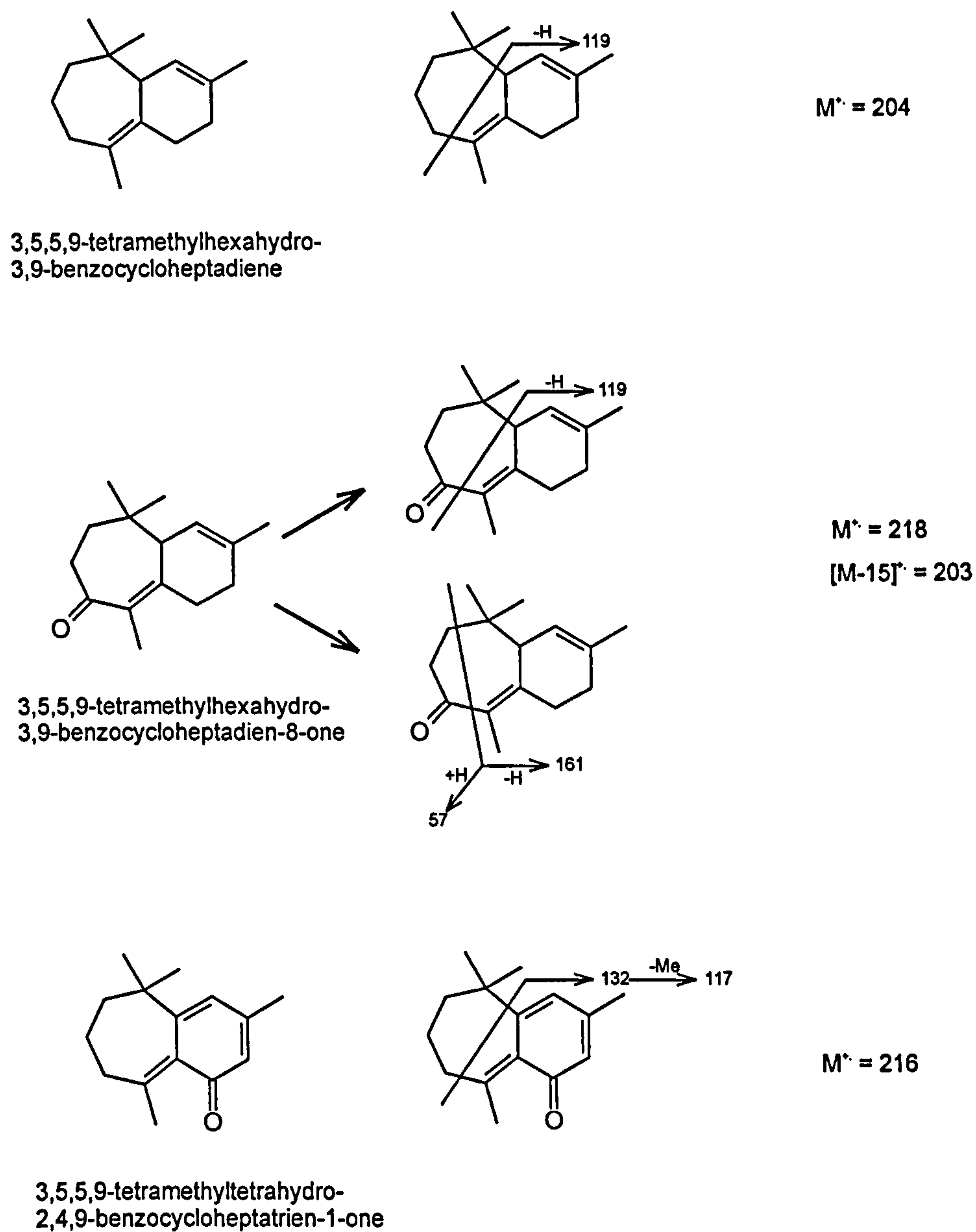


Figure 6.6 The major mass fragmentations of the benzocycloheptenes identified in the TD-GC/MS and GC/MS analyses, and the reaction mechanism (autoxidation) for the formation of the 8-one (the 1-one would also be expected to be formed) compound (Scheme 1) from the blackened wrapping from the base of the mummy [1] of the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

predominating were observed, as were branched chain fatty acids ($C_{15:0}$ and $C_{17:0}$) as minor components. In addition, the mono- and dihydroxy fatty acids were detected in similar abundance to the branched chain fatty acids. Moderate amounts of the aromatic acids hydroxybenzoic, vanillic and terephthalic acids were also identified. The diterpenoids, dehydroabietic, 7-oxodehydroabietic and 15-hydroxy-7-oxodehydroabietic acids (see Fig. 6.4) were minor components of the acid fraction, with comparable quantities of triterpenoid acids. This latter group included three dammarane acids (e.g. shoreic acid) and based on both retention times and mass spectra these dammarane/octillone-type triterpenoids were tentatively identified as 20,24-epoxy-25-hydroxy-3,4-seco-4(28)-dammaran-3-oic acids [as their free hydroxy (due to steric hindrance), TMS esters; two isomers] and its hydrated derivative 20,24-epoxy-4,25-dihydroxy-3,4-secodammaran-3-oic acid [as the free monohydroxy (25 position), mono-TMS ether, TMS ester; again due to steric hindrance of the tertiary hydroxyl group] (see Fig. 6.4; Chapter 5, Fig. 5.11). The compounds were characterised by m/z 125 and 143 (base peak). As with 'Neskhons' the presence of these compounds is tentatively supported by the TD analysis where 20,24-epoxy-28(?)-nordammar-25-en-3-one (two isomers) was detected, apparently being formed in the probe at 310°C via the cyclisation (loss of HCHO and H₂O) and dehydration of these acids. The relative abundances of these compounds would seem to agree with this. Notably, the dammaranes were not as highly oxidised as those detected in the wrappings of the XXIInd dynasty mummy, Neskhons (5.4.7.1 and 5.4.7.2). Again unlike Neskhons, moronic acid (TMS ester, characterised by m/z 73, 189 and 203; Fig. 6.4) was identified as the major triterpenoid acid along with 11-hydroxyoleanonic acid (TMS ether, TMS ester, characterised by m/z 73, 161, 189, 203, 279 and 309). Unfortunately, the abundances of some of these triterpenoid acids were too low to obtain good mass spectra which could aid their positive identification.

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 6.3c. A series of *n*-alkanes (C_{23} to C_{33} maximising at C_{27}) with an odd-over-even preference were observed, along with wax esters in the C_{38} to C_{50} carbon number range, with C_{42} predominating; these wax esters were the major constituents of the neutral fraction. The C_{38} to C_{48} wax esters contained predominantly the $C_{16:0}$ acyl group with lesser amounts of the $C_{18:0}$, and the C_{50} predominantly $C_{18:0}$ with significant amount of the $C_{16:0}$ acyl group. Monohydroxy wax esters (C_{44} to C_{48}) were present as trace components, characterised by m/z 117 and 329, and eluting just after the wax esters of corresponding carbon number.

Eluting just after the wax esters, four triacylglycerols, C₄₈ tri-C_{16:0}/C_{16:0}/C_{16:0}-glycerol, C₅₀ tri-C_{16:0}/C_{16:0}/C_{18:0}-glycerol, C₅₂ tri-C_{16:0}/C_{18:0}/C_{18:0}-glycerol and C₅₄ tri-C_{18:0}/C_{18:0}/C_{18:0}-glycerol were observed as significant components. In addition, the two triterpenoids, nor- α -amyrone [m/z 189 and 204 (base peak)] and nor- β -amyrone [m/z 189 and 204 (base peak)] were also present as significant constituents, with lesser amounts of the C_{16:0} and C_{18:0} 1-monoacylglycerols and methyl 7-oxodehydroabietate (although retene was notably absent). The monoterpenoid thymol and a number of sesquiterpenoids were also observed eluting between 0.5 and 4.0 min. The sesquiterpenoids included two cadinenes (cadinatrienes, with one aromatised ring), and two oxidised substituted benzocycloheptenes, all of which were also seen in the TD profile, confirming that they were not thermolytically derived during TD. The two oxidised sesquiterpenoids were identified as 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadien-8-one, characterised by m/z 91, 105, 119 (base peak), 161, 203 and M^+ 218 and 3,5,5,9-tetramethyl-tetrahydro-2,4,9-benzocycloheptatrien-1-one, characterised by m/z 91, 117, 132 (base peak) and M^+ 216 (see Figs. 6.4 & 6.5). Their identification was also based on their retention times and the presence of the free, naturally occurring tetramethyl-hexahydro-benzocycloheptadiene in the TD (see discussion below); this compound was absent from the neutral fraction due to its high volatility causing it to be lost during sample handling, e.g. rotary evaporation and blowing down.

6.4.3.2. Detached 'resin'-soaked wrapping 1 [2].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 6.7a. The TD profile was very similar to 6.4.3.1 displaying a complex pattern with many components present. A series of monocarboxylic acids (C₇ to C₁₈) were detected, the major components being C_{16:0}, and C_{18:0} in decreasing order of abundance, these last two fatty acids dominating the TD profile. Two C_{18:2} fatty acids were also identified as minor components, with a trace amount of C_{18:1} and unusually high relative abundances of the short chain fatty acids (C_{7:0} to C_{9:0}), suggesting that the original oil or fat had undergone appreciable oxidative change. The steroidal compound, cholesta-3,5,7-triene was detected. A series of *n*-alkanes, C₂₅ to C₃₁, with an odd-over-even preference were observed as minor components, as were the diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate. The monoterpenoid, thymol was present in low abundance, along with more appreciable amounts of bicyclic sesquiterpenoids which included a number cadinenes and several substituted benzocycloheptenes. The major cadinenes identified were two cadinatetraenes,

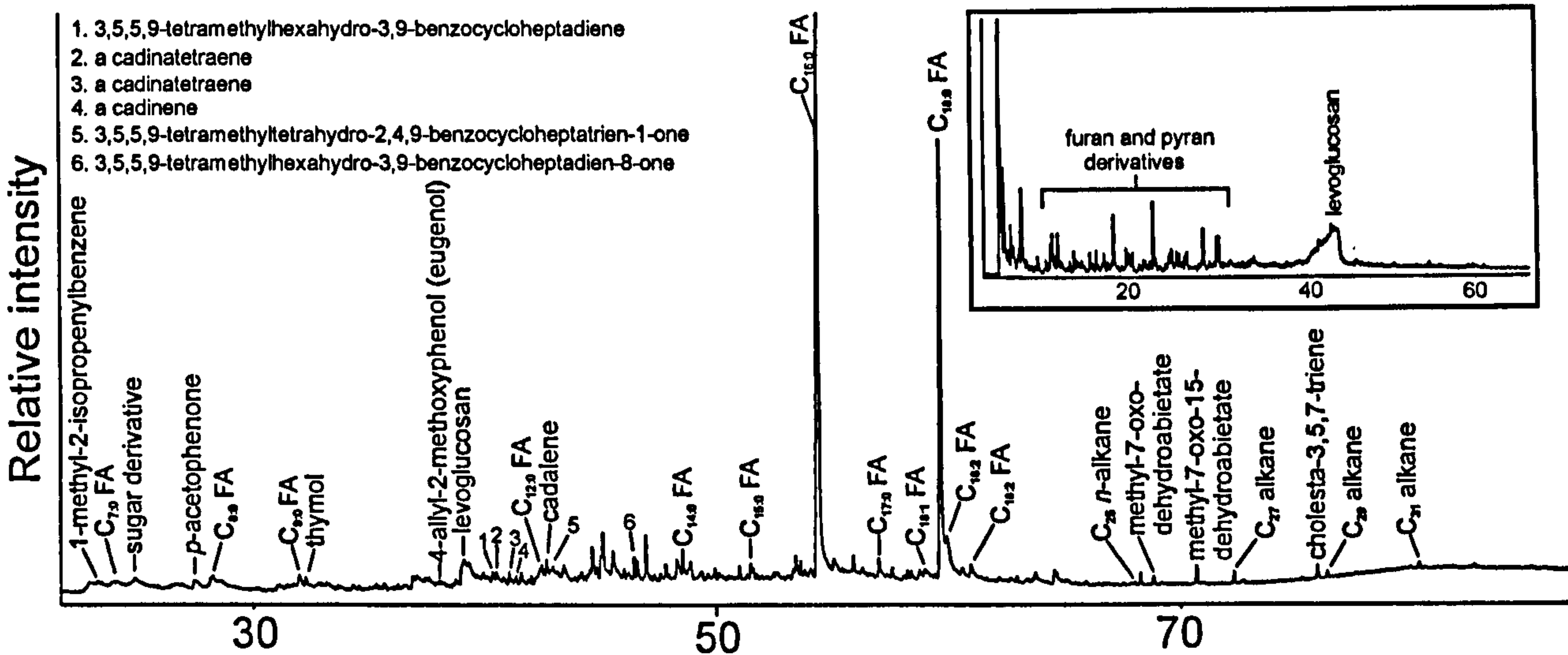


Figure 6.7a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of detached 'resin'-soaked wrapping 1 [2] from the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

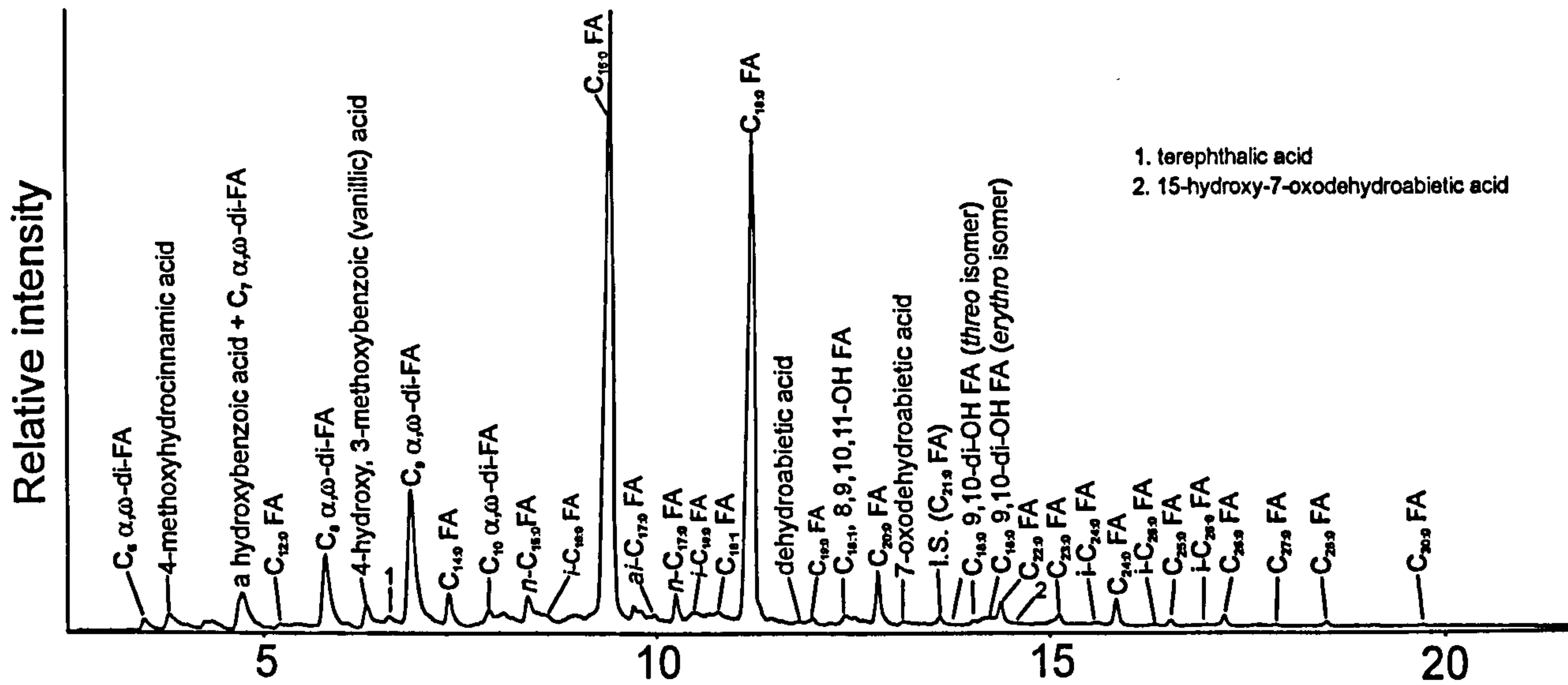


Figure 6.7b Total ion chromatogram from the GC/MS analysis of the acid fraction of detached 'resin'-soaked wrapping 1 [2] from the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

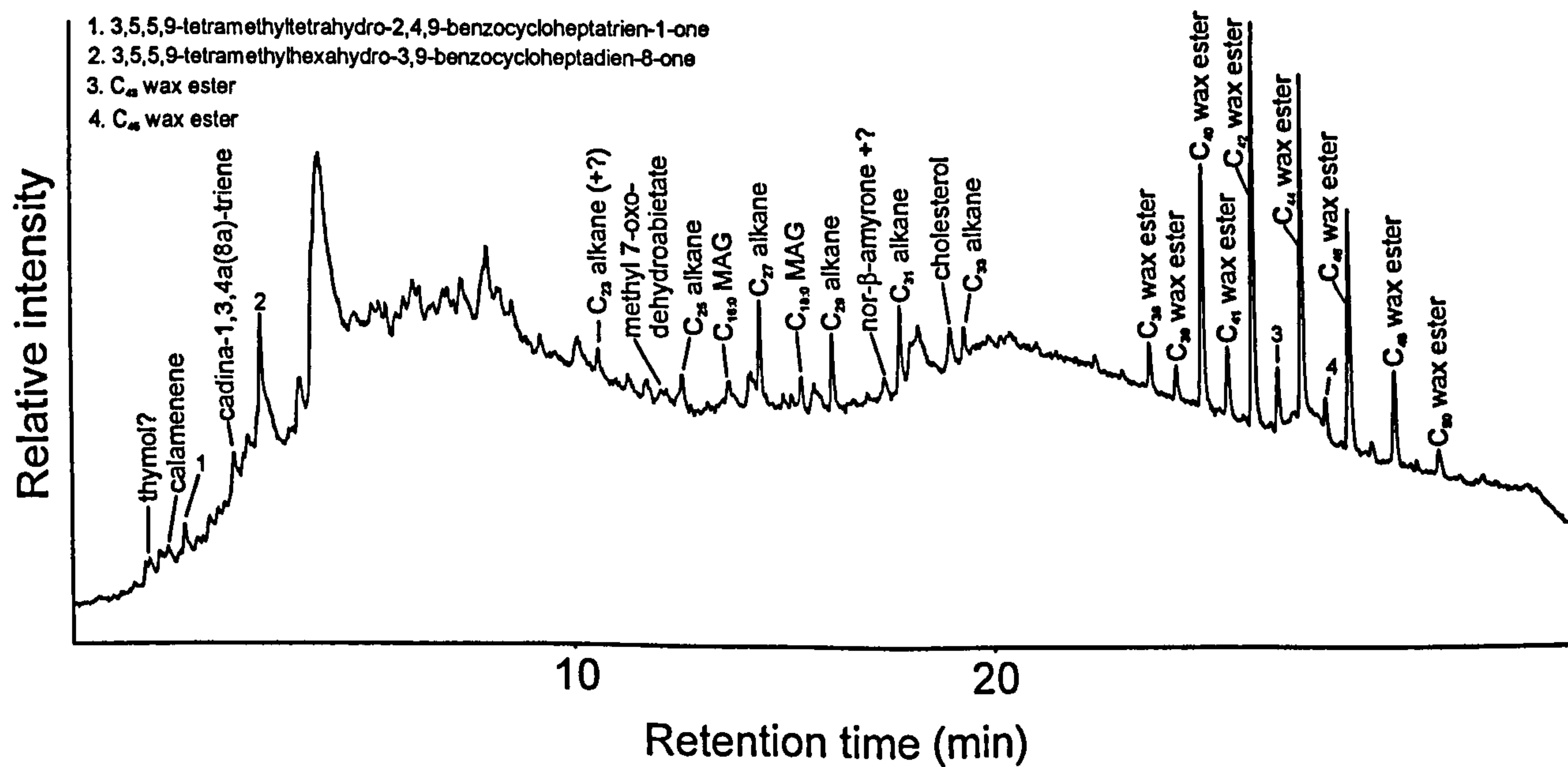


Figure 6.7c Total ion chromatogram from the GC/MS analysis of the neutral fraction of detached 'resin'-soaked wrapping 1 [2] from the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

two cadinatrienes and the fully aromatised cadalene. The two oxidised sesquiterpenoids were identified as 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadien-8-one and 3,5,5,9-tetramethyl-tetrahydro-2,4,9-benzocycloheptatrien-1-one. These two compounds were accompanied by the naturally occurring (see discussion) 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadiene. Eugenol was present as a trace component, although caryophyllene (along with the associated triterpenoids), observed in 6.4.3.1, was not detected in this sample. Levoglucosan and furan derivatives were present as minor components, suggesting a possible sugar/plant gum origin, and this is supported by the presence of cadinenes, however, the sugar markers may derive from hydrolysed cellulose present in the degraded linen wrappings (see discussion).

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 6.7a, inset. Although the pyrogram was not studied in detail. The main components (with the exception of CO₂) were levoglucosan and the furan and pyran derivatives, although these almost certainly derive from the cellulose which constitutes the linen wrappings. The fatty acids present in the pyrogram for the previous sample (6.4.3.1) were not observed, indicating that the fatty acids in this previous sample (6.4.3.1) were present in a polymeric/bound form.

GC/MS – Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 6.7b. The major components were monocarboxylic acids (C₁₂ to C₃₀), with C_{16:0} and C_{18:0} predominating. The C_{18:1} unsaturated acid was present as a minor constituent of the sample, as were saturated long-chain fatty acids in the C_{22:0} and C_{30:0} carbon number range with C_{24:0} predominating and with trace quantities of the *i*-C_{24:0}, *i*-C_{25:0} and *i*-C_{26:0} fatty acids. Significant quantities of α,ω -dicarboxylic acids (C₆ to C₁₁), with C₈ and C₉ predominating were observed, as were branched chain fatty acids (C_{15:0} and C_{17:0}) as minor components. In addition, the mono- and dihydroxy fatty acids were detected in similar abundance to the branched chain fatty acids. Moderate amounts of the aromatic acids hydroxybenzoic, vanillic and terephthalic acids were also identified. The diterpenoids, dehydroabietic, 7-oxodehydroabietic and 15-hydroxy-7-oxodehydroabietic acids were minor components of the acid fraction, but the triterpenoid acids present in the previous sample were not detected.

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 6.7c. A series of *n*-alkanes (C₂₃ to C₃₃ maximising at C₂₇) with an odd-over-even preference were observed, along with wax esters in the C₃₈ to C₅₀ carbon number range, with C₄₂ predominating; these wax esters were the major constituents of the neutral fraction. The C₃₈, C₄₀, C₄₂ and C₄₄ wax esters contained predominantly the C_{16:0} acyl group with lesser amounts of the C_{18:0}, with the C₄₆, C₄₈ and C₅₀ consisting predominantly of C_{18:0} with significant amounts of the C_{16:0} acyl group. Monohydroxy wax esters (C₄₄ to C₄₈) were present as trace components, characterised by *m/z* 117 and eluting just after the wax esters of corresponding carbon number. The triterpenoid, nor- β -amyrone was also present as a minor constituent, as was cholesterol, with similar amounts of the C_{16:0} and C_{18:0} 1-monoacylglycerols and methyl 7-oxodehydroabietate. The monoterpenoid thymol and a number of sesquiterpenoids were also observed. The sesquiterpenoids were the same constituents identified in sample 6.4.3.1 and included two cadinenes (cadinatrienes, with one aromatised ring), and two oxidised substituted benzocycloheptenes, all of which were also seen in the TD profile. The two oxidised sesquiterpenoids were identified as 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadien-8-one and 3,5,5,9-tetramethyl-tetrahydro-2,4,9-benzocycloheptatrien-1-one. Their identification was also based on their retention times and the presence of the free, naturally occurring tetramethyl-hexahydro-benzocycloheptadiene in the TD (see discussion below). The triacylglycerols observed in sample 6.4.3.1 were absent from this sample.

6.4.3.3 Detached 'resin'-soaked wrapping 2 [3]

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 6.8a. The TD profile was very similar to 6.4.3.1 & 6.4.3.2 displaying a complex pattern with many peaks present. The TD profile displayed a fairly complex pattern with many peaks present. A series of monocarboxylic acids (C₇ to C₁₈) were detected, the major components being C_{16:0}, and C_{18:0} in decreasing order of abundance, these last two fatty acids dominating the TD profile. The C_{18:1} and short chain fatty acids (C_{7:0} to C_{9:0}) were present as a minor components, with two C_{18:2} fatty acids identified as trace components. The steroidal compound, cholesta-3,5,7-triene was also detected. A series of *n*-alkanes, C₂₅ to C₃₁, with an odd-over-even preference were observed as trace components, as were the diterpenoids methyl 7-oxodehydroabietate and methyl 7-oxo-15-dehydrodehydroabietate, along with

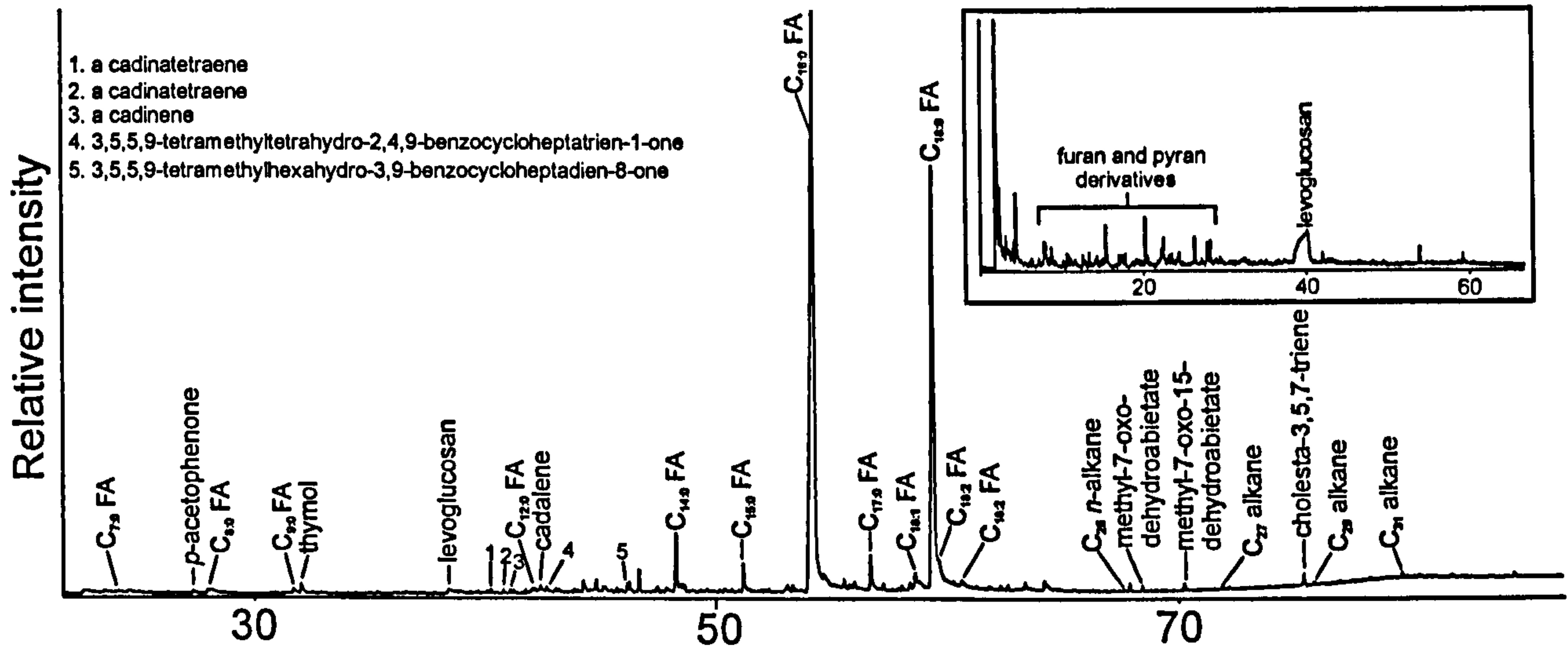
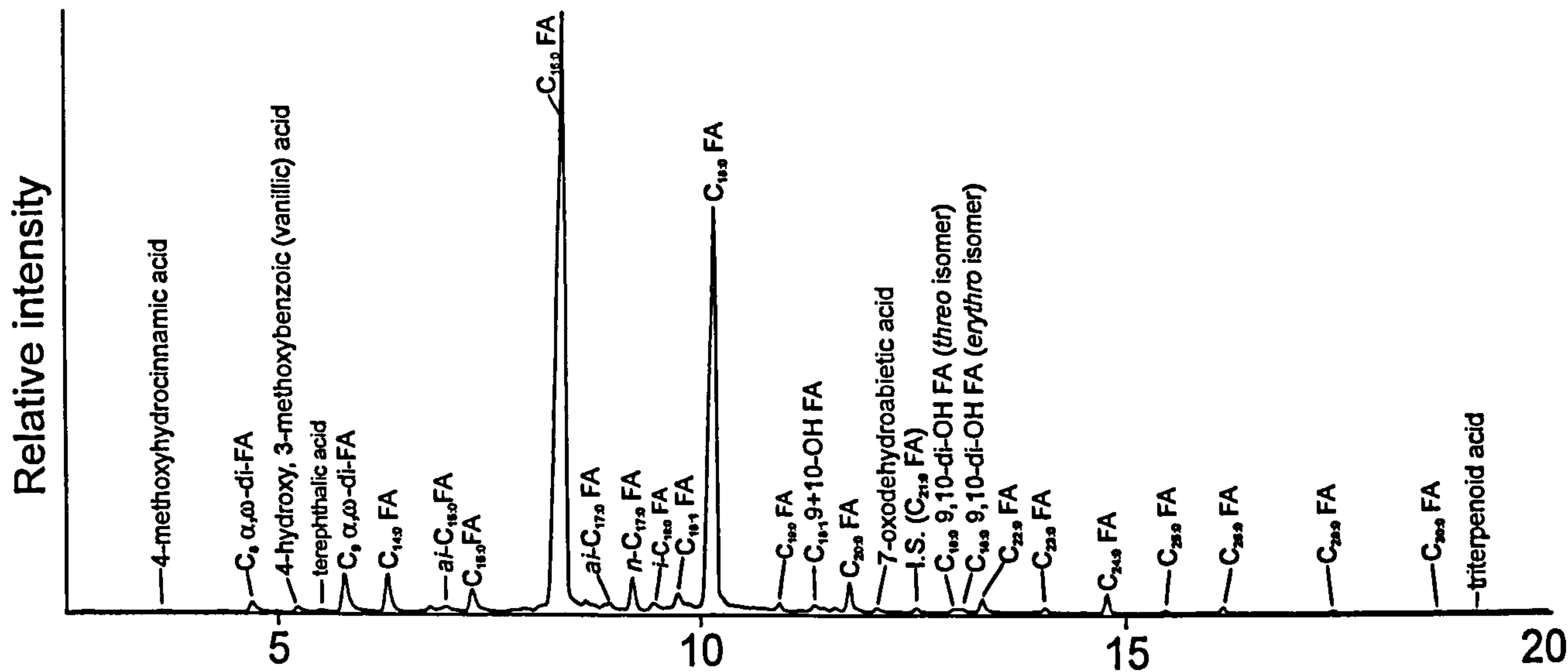


Figure 6.8a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of detached 'resin'-soaked wrapping 2 [3] from the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).



more appreciable amounts of bicyclic sesquiterpenoids which included a number of cadinenes and several substituted benzocycloheptenes. The major cadinenes were identified as two cadinatetraenes, two cadinatrienes and the fully aromatised cadalene. The two oxidised sesquiterpenoids were identified as 3,5,5,9-tetramethyl-hexahydro-3,9-benzocyclo-heptadien-8-one and 3,5,5,9-tetramethyl-tetrahydro-2,4,9-benzocyclo-heptatrien-1-one. Again caryophyllene was not detected in this sample. Levoglucosan and furan derivatives were detected in trace quantities, suggesting a possible sugar/plant gum origin, and this is supported by the presence of cadinenes. However, the sugar markers may also derive from hydrolysed cellulose present in the degraded linen wrappings (see discussion). Triterpenoids also were observed, albeit as trace components.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 6.8a, inset. The main components (with the exception of CO₂) were levoglucosan and the furan and pyran derivatives, although these almost certainly derive from the cellulose which constitutes the linen wrappings. The fatty acids observed in the pyrogram for the previous sample (6.4.3.1) were observed only as minor components, supporting the suggestion that the fatty acids in the previous sample were present in a polymeric/bound form.

GC/MS – Acid fraction

The results for the acid fraction analysed by GC/MS are shown in Figure 6.8b. The major components were monocarboxylic acids (C₁₄ to C₃₀), with C_{16:0} and C_{18:0} predominating. The C_{18:1} unsaturated acid was present as a minor constituent of the sample, as were saturated long-chain fatty acids in the C_{22:0} and C_{30:0} carbon number range with C_{24:0} predominating. α,ω -Dicarboxylic acids (C₈ to C₁₀), were observed in moderate abundance, while branched chain fatty acids (C_{15:0} and C_{17:0}) were minor components. In addition, mono- and dihydroxy fatty acids were detected in similar abundance to the branched chain fatty acids. The aromatic acids vanillic and terephthalic acids were also identified in low abundance, as was the diterpenoid, 7-oxodehydroabietic acids. Coeluting at 19 min, two triterpenoid acids were detected albeit as trace constituents, a dammarane/ocotillone component, characterised by *m/z* 125 and 143 (base peak) and a hydroxyoleanonic acid (TMS ether, TMS ester), characterised by *m/z* 189 and 203). Unfortunately, the abundances of the triterpenoid acids were too low to obtain good mass spectra which could aid their positive identification.

GC/MS – Neutral fraction

The results for the neutral fraction analysed by GC/MS are shown in Figure 6.8c. A series of *n*-alkanes (C_{23} to C_{33} maximising at C_{31}) with an odd-over-even preference were observed, along with wax esters in the C_{38} to C_{50} carbon number range, with C_{42} predominating; these wax esters were the major constituents of the neutral fraction. The C_{38} to C_{50} wax esters contained predominantly the $C_{16:0}$ acyl group with lesser amounts of the $C_{18:0}$. Eluting just after the wax esters, four triacylglycerols, C_{48} tri- $C_{16:0}/C_{16:0}/C_{16:0}$ -glycerol, C_{50} tri- $C_{16:0}/C_{16:0}/C_{18:0}$ -glycerol, C_{52} tri- $C_{16:0}/C_{18:0}/C_{18:0}$ -glycerol and C_{54} tri- $C_{18:0}/C_{18:0}/C_{18:0}$ -glycerol were also observed as minor components. The triterpenoid, nor- β -amyrene was present as a minor constituent, as was cholesterol and methyl 7-oxo-dehydroabietate. Two sesquiterpenoids were also observed, these being two cadinenes (cadinatrienes, with one aromatised ring), and 3,5,5,9-tetramethyltetrahydro-2,4,9-benzocycloheptatrien-1-one, all of which were also seen in the TD profile.

6.4.3.4 Red material in right ear [5]

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 6.9a. The TD profile contained a number of carbohydrate markers, including levoglucosan (m/z 43, 57, 60 (base peak) 73 and 98) with comparable amounts of furan and pyran derivatives. These included 3-hydroxy-2-methyl-(4H)-pyranone (2 isomers) (m/z 43, 55, 71, 97 and M^+ 126 (base peak), 5-hydroxymethyl-2-tetrahydrofuraldehyde-3-one (2 isomers) (m/z 41, 43 (base peak), 57, 69, 70, 85 and M^+ 144), 1,4:3,6-dianhydro- α -D-glucopyranose (m/z 41, 57, 69 (base peak) 98 and 144) and 1,6-anhydro- β -D-glucofuranose (m/z 43, 44, 61, 69, 73 (base peak) and 85). Their presence suggests a possible sugar/plant gum origin, particularly given the absence of any cellulose based wrappings in this sample. The $C_{16:0}$ and $C_{18:0}$ fatty acids were also significant components, with minor quantities of C_{25} to C_{31} alkanes, maximising at C_{27} , with an odd-over-even preference.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 6.9a, inset. The pyrogram displayed levoglucosan, as the major constituent, and the furan and pyran derivatives seen in the TD; these are presumed to have derived from the sugar/gum, only partially volatilised and/or trapped in the interface at 310°C/10s.

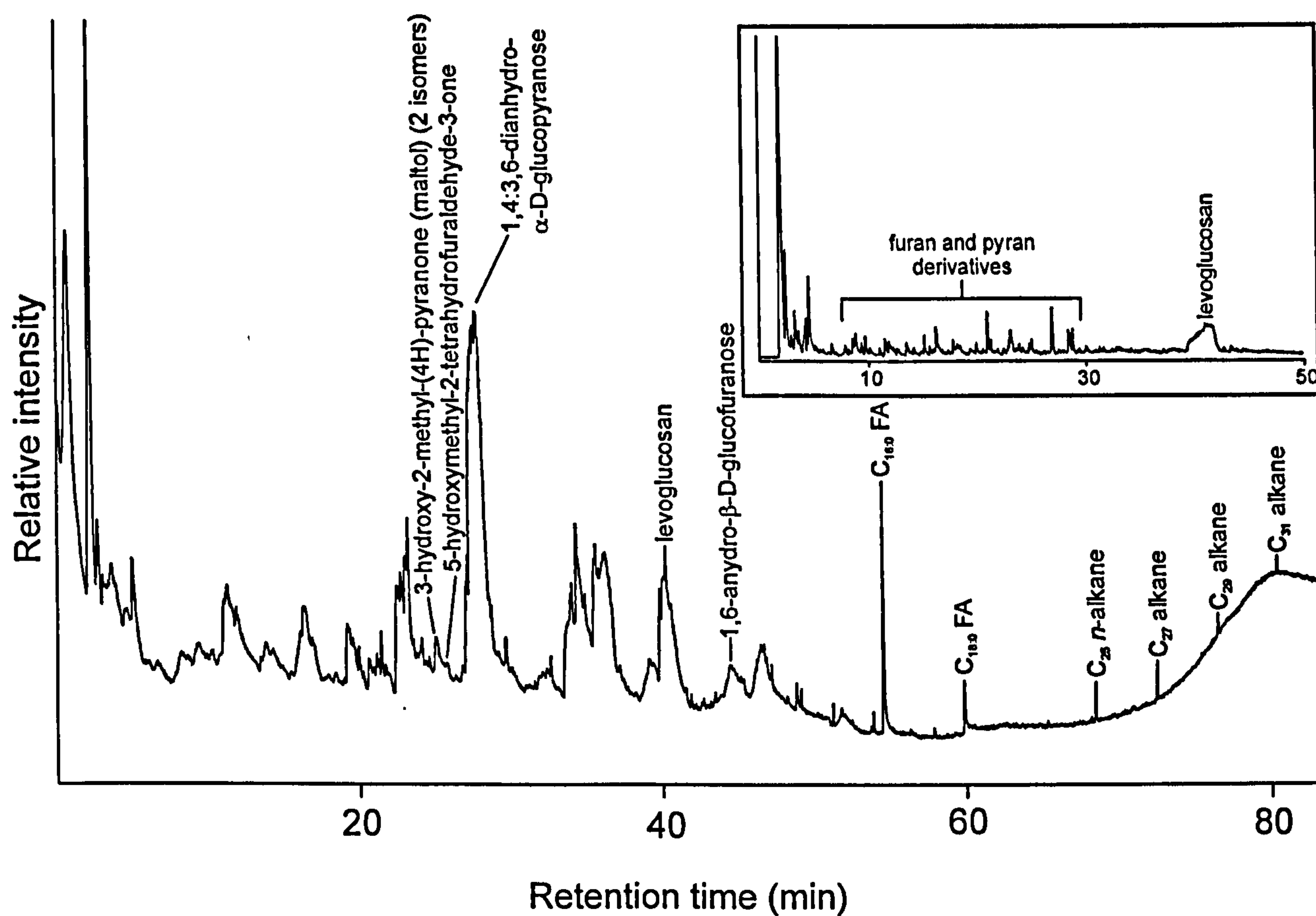


Figure 6.9a Total ion chromatograms of the thermal desorption profile (310°C/10s), and (inset) the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of the red material in the right ear [5] of the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

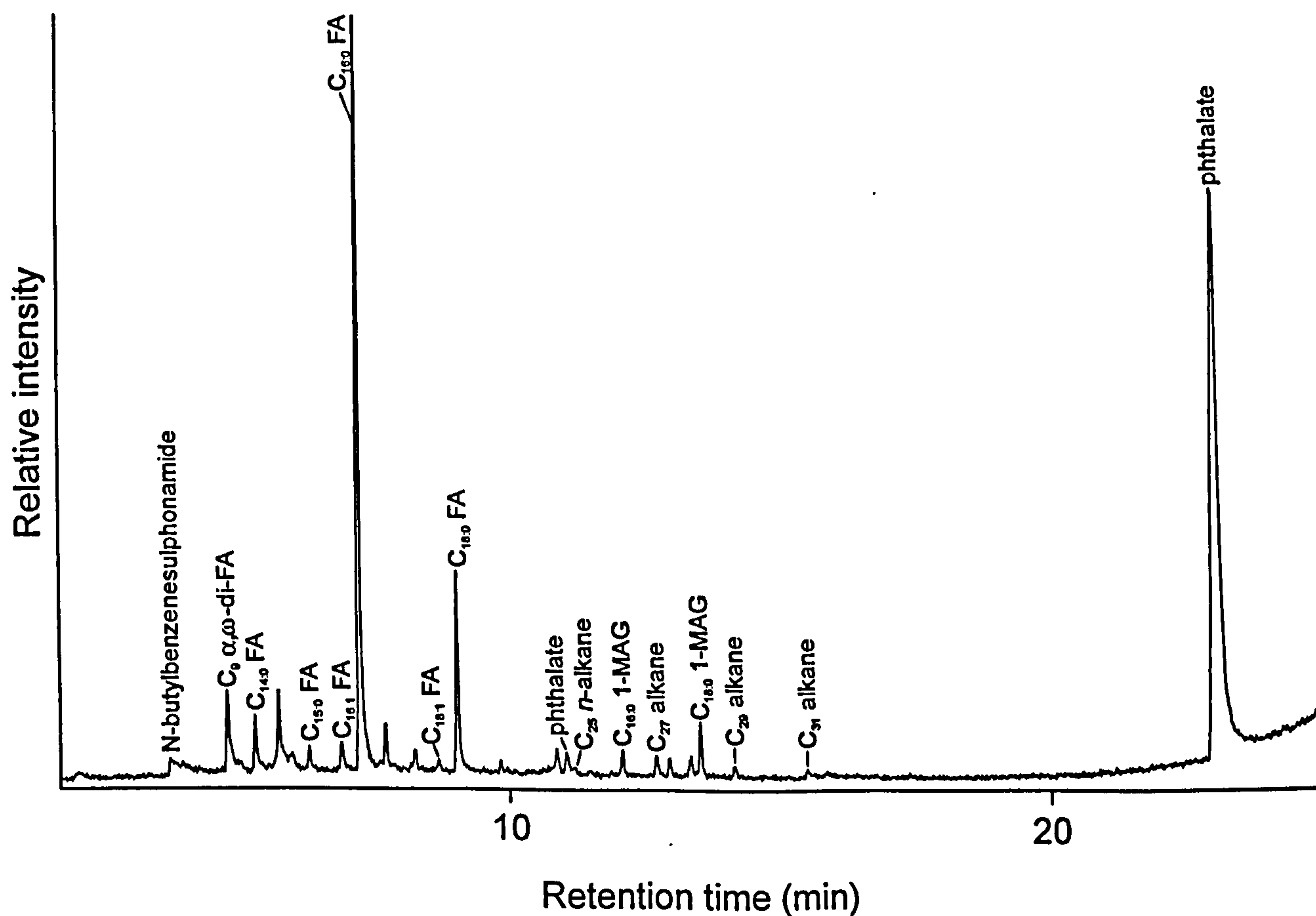


Figure 6.9b Total ion chromatogram from the GC/MS analysis of the total lipid extract of the red material in the right ear [5] of the Late Period cat, XXVIth-XXXth dynasty (c. 664-343 B.C.).

GC/MS – Total lipid extract

The results of the GC/MS total lipid extract analysis are shown in Figure 6.9b. Monocarboxylic acids (C_{14} to C_{18}) were identified with $C_{16:0}$ and $C_{18:0}$ being the main two components in decreasing order of abundance. The C_9 α,ω -dicarboxylic acid was a significant constituent of the extract, as were the $C_{16:0}$ and $C_{18:0}$ 1-monoacylglycerols. The $C_{16:1}$ and $C_{18:1}$ unsaturated acids were present as trace constituents of the sample, as were the C_{25} to C_{31} *n*-alkanes, maximising at C_{27} with an odd-over-even preference. Wax esters which would have been anticipated to be associated with the alkanes were not detected. The second most abundant constituent of the extract was in fact a phthalate, a common plasticiser and contaminant reflecting the fact that very little extractable material was present.

6.4.4 Ibis, XXVIth–XXXth dynasty (c.664-343 BC), Sakkarā (1969.112.42)

6.4.4.1 'Resin'-soaked wrapping covering right breast [1].

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 6.10a. The TD profile was dominated by levoglucosan [m/z 43, 57, 60 (base peak) 73 and 98] and less abundant furan and pyran derivatives. These included 3-hydroxy-2-methyl-(4H)-pyranone [2 isomers; m/z 43, 55, 71, 97 and M^+ 126 (base peak)], 5-hydroxymethyl-2-tetrahydrofuraldehyde-3-one [2 isomers; m/z 41, 43 (base peak), 57, 69, 70, 85 and M^+ 144], 1,4:3,6-dianhydro- α -D-glucopyranose [m/z 41, 57, 69 (base peak) 98 and 144], 5-hydroxymethyl-2-furaldehyde [m/z 39, 41, 53, 69, 97 (base peak) and M^+ 126] and 1,6-anhydro- β -D-glucofuranose [m/z 43, 44, 61, 69, 73 (base peak) and 85]. Their presence suggests a possible sugar/plant gum origin, although they may derive from hydrolysed cellulose present in the degraded linen wrappings. As shown in the two XVIIth dynasty human mummies, undegraded cellulose does not produce levoglucosan and the furan and pyran derivatives at the TD desorption temperatures used in this study, breakdown of the cellulose only taking place at the 610°C employed for pyrolysis.

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 6.10b. The pyrogram was not studied in detail, but displayed levoglucosan, as the major constituent, and the furan and pyran derivatives seen in the TD. However, three additional components were also present as major constituents of the pyrolysate, i.e. 2-hydroxy-2-cyclopenten-1-one [m/z 41, 42,

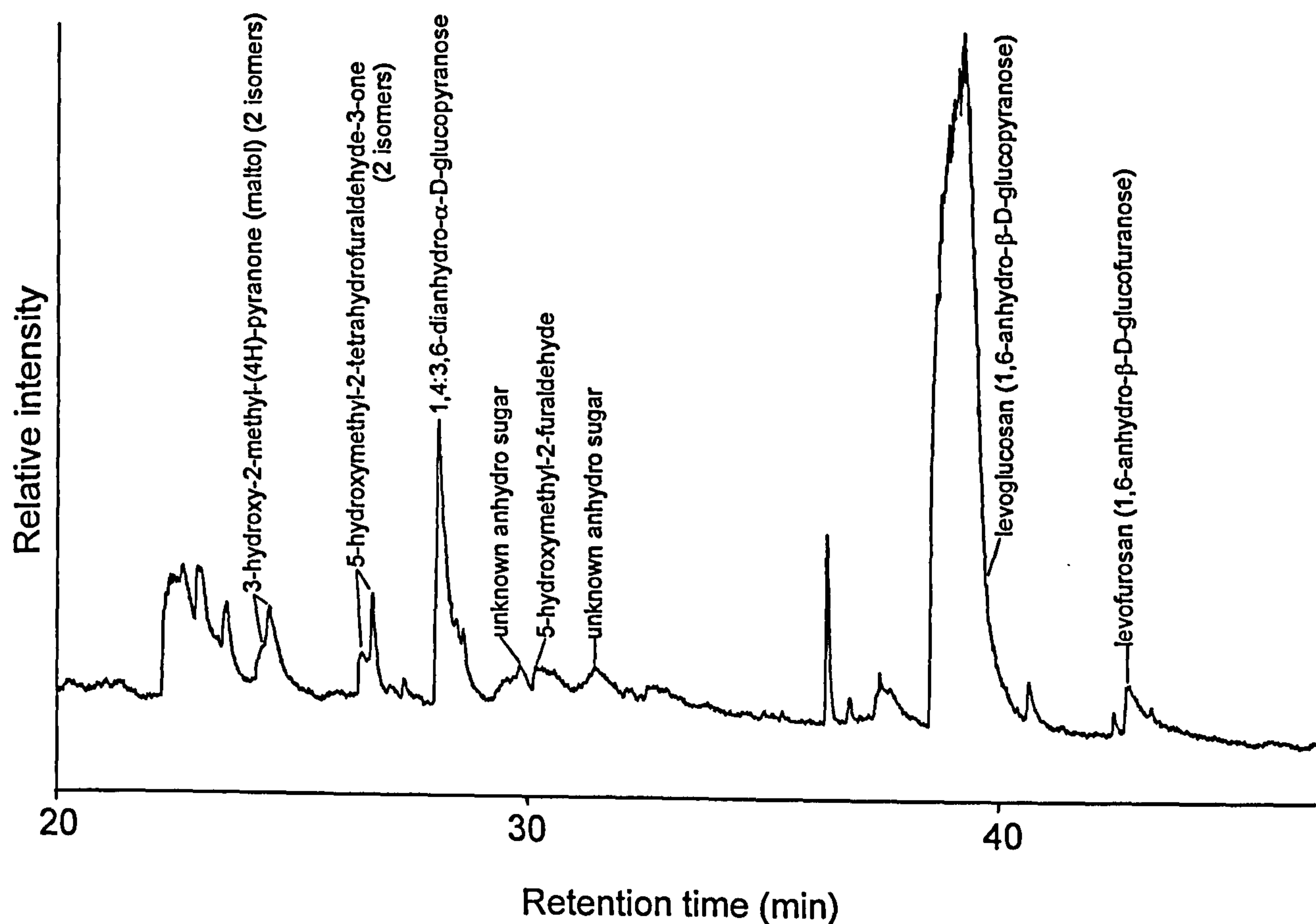


Figure 6.10a Total ion chromatogram of the thermal desorption profile (310°C/10s) of 'resin'-soaked wrapping covering the right breast [1] of the Late Period ibis, XXVIth-XXXth dynasty (c. 664-343 B.C.).

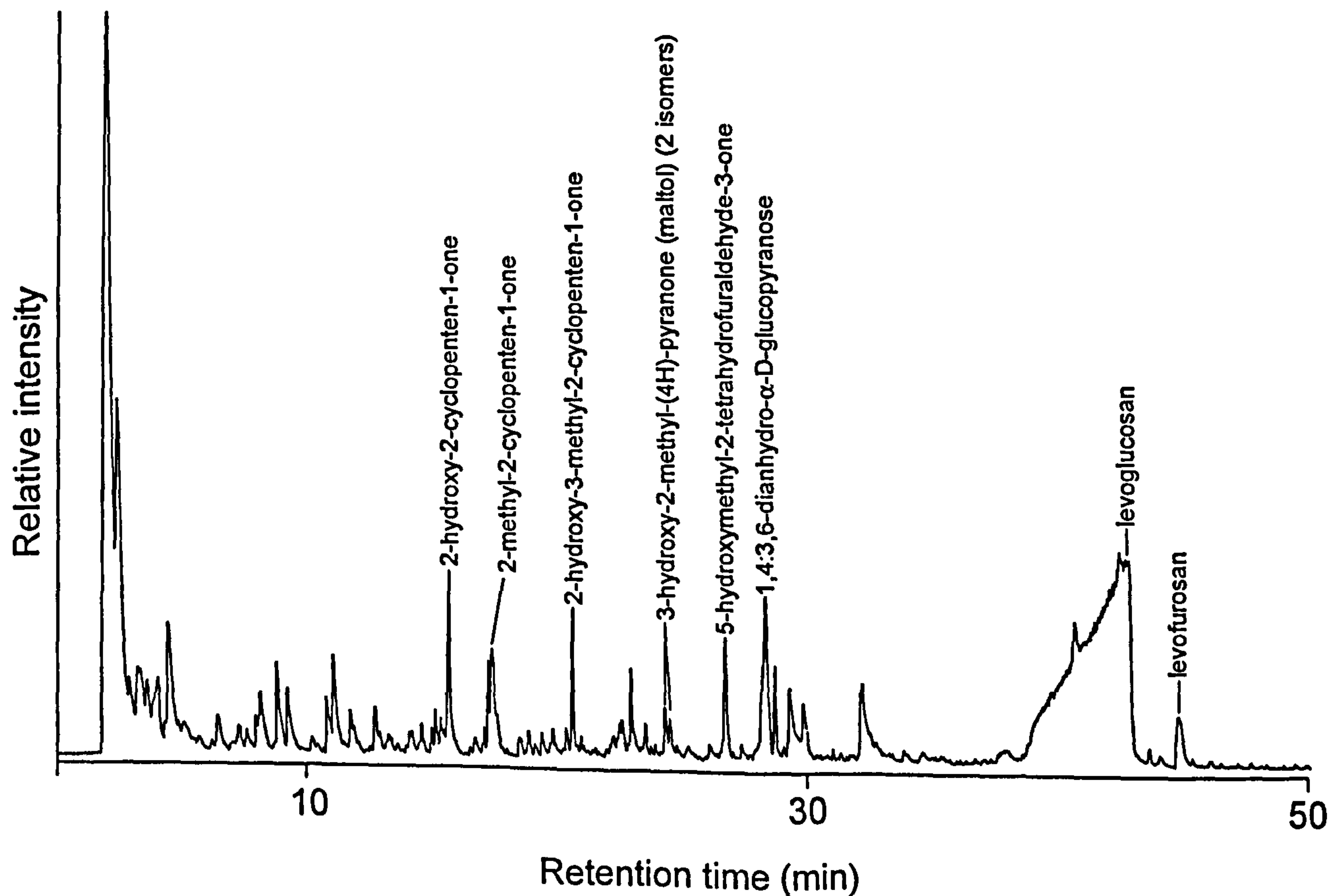


Figure 6.10b Total ion chromatogram of the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin'-soaked wrapping covering the right breast [1] of the Late Period ibis, XXVIth-XXXth dynasty (c. 664-343 B.C.).

55, 69, 70 and 98 (base peak)], 2-methyl-2-cyclopenten-1-one [m/z 41, 53, 67, 68 and 96 (base peak)] and 2-hydroxy-3-methyl-2-cyclopenten-1-one [m/z 55, 56, 69, 83, 84 and 112 (base peak)]; these were absent from the TD profile] which are major compounds in the pyrolysate of cellulose (Faix et al. 1991, p.213-219; Faix et al. p.299-304; Galletti & Bocchini 1995, p.815-826). These cyclopenten-1-one derivatives, along with maltol (3-hydroxy-2-methyl-(4H)-pyranone) (2 isomers), 5-hydroxymethyl-2-tetrahydrofuraldehyde-3-one (1 isomer), 1,4:3,6-dianhydro- α -D-glucopyranose, levoglucosan (1,6-anhydro- β -D-glucopyranose) and 1,6-anhydro- β -D-glucofuranose almost certainly derive from the cellulose which constitutes the linen wrappings.

GC/MS - Total lipid extract

There was very little extractable material in this sample, the results for the 'total lipid extracts' analysed by GC/MS displaying only trace amounts of the $C_{16:0}$ monocarboxylic acid (as its TMS ester) and the C_{44} , C_{46} , and C_{48} wax esters. Alkanes, which may have been associated with the wax esters, were not detected, as would be expected given the small amount of extractable material present. The main component of the extract was the phthalate contaminant observed in the other animal mummies.

6.4.4.2 'Resin'-soaked wrapping covering left breast [2]

TD-GC/MS

The results of the TD-GC/MS analysis are shown in Figure 6.11a. The TD profile was dominated by levoglucosan and lesser amounts of furan and pyran derivatives. These included 3-hydroxy-2-methyl-(4H)-pyranone (2 isomers), 5-hydroxymethyl-2-tetrahydrofuraldehyde-3-one (2 isomers), 1,4:3,6-dianhydro- α -D-glucopyranose, 5-hydroxymethyl-2-furaldehyde and 1,6-anhydro- β -D-glucofuranose. Their presence suggests a possible sugar/plant gum origin, particularly given their appreciable abundances, although they may derive from hydrolysed cellulose present in the degraded linen wrappings. As shown in the two XVIIth dynasty human mummies, undegraded cellulose does not produce levoglucosan and the furan and pyran derivatives at the TD desorption temperatures used in this study, breakdown of the cellulose only taking place at the 610°C employed for pyrolysis. The TD profile was very similar to the previous ibis sample (6.4.4.1), but also contained low abundances of the cyclohexane derivatives tetradecylcyclohexane, pentadecylcyclohexane and hexadecylcyclohexane in increasing order of abundance.

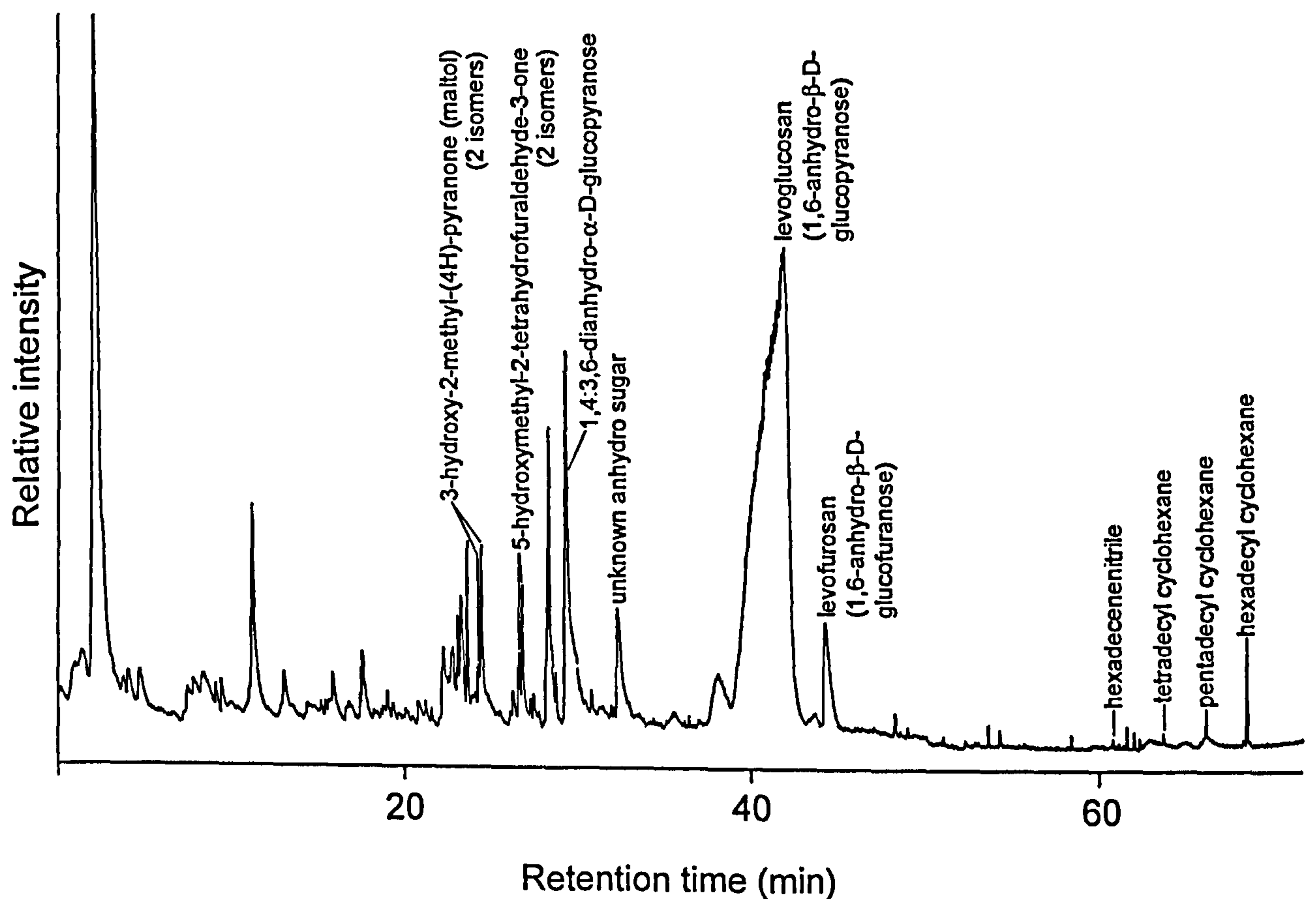


Figure 6.11a Total ion chromatogram of the thermal desorption profile (310°C/10s) of 'resin'-soaked wrapping covering the left breast [2] of the Late Period ibis, XXVIth-XXXth dynasty (c. 664-343 B.C.).

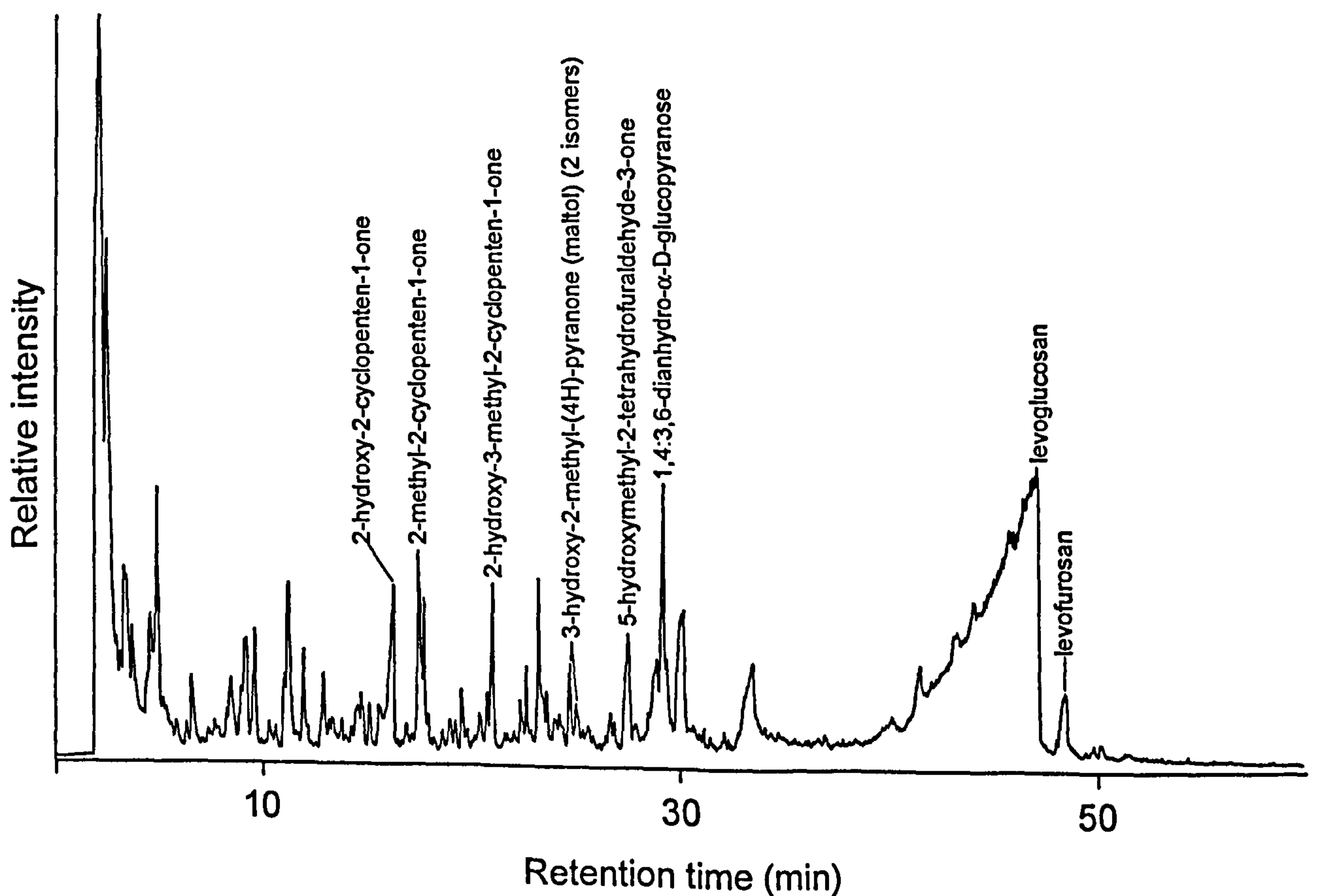


Figure 6.11b Total ion chromatogram of the pyrolysis profile (610°C/10s, after TD at 310°C/10s) of 'resin'-soaked wrapping covering the left breast [2] of the Late Period ibis, XXVIth-XXXth dynasty (c. 664-343 B.C.).

Py-GC/MS

The results of the Py-GC/MS analysis are shown in Figure 6.11b. The pyrogram was not studied in detail, but displayed levoglucosan, as the major constituent, and the furan and pyran derivatives seen in the TD. However, three additional components were also present as major constituents of the pyrolysate, i.e. 2-hydroxy-2-cyclopenten-1-one, 2-methyl-2-cyclopenten-1-one and 2-hydroxy-3-methyl-2-cyclopenten-1-one (these were absent in the TD profile) which, as mentioned above, are major compounds in the pyrolysate of cellulose. These cyclopenten-1-one derivatives, along with maltol (3-hydroxy-2-methyl-(4H)-pyranone) (2 isomers), 5-hydroxymethyl-2-tetrahydrofural dehyde-3-one (1 isomer), 1,4:3,6-dianhydro- α -D-glucopyranose, levoglucosan (1,6-anhydro- β -D-glucopyranose) and 1,6-anhydro- β -D-glucofuranose almost certainly derive from the cellulose which constitutes the linen wrappings.

GC/MS-Total lipid extract

There was very little extractable material in this sample, the results for the 'total lipid extracts' analysed by GC/MS displaying only trace amounts of the $C_{16:0}$ monocarboxylic acid (as its TMS ester) and the C_{29} , C_{31} , C_{34} and C_{36} alkanes, with the most abundant components being monosaccharides (as their TMS derivatives). These sugars included a furanose, characterised by m/z 73, 147, 191, 204 and 217, and two pyranoses, also characterised by m/z 73, 147, 191, 204 and 217. However, in the furanoses m/z 217 is the most abundant of the 191, 204 and 217 peaks, due to the formation of $(CH_3)_3SiOCH=CHCH=O^+Si(CH_3)_3$, whereas in pyranoses m/z 204 is the dominant peak, due to the formation of $[(CH_3)_3SiOCH=CHOSi(CH_3)_3]^+$; more abundant in the pyranoses due to the greater number of OH groups on adjacent carbon atoms (Pierce, 1968, p.37-38). Wax esters, which would have been expected to have been associated with the alkanes, were not detected. The main component of the extract was the phthalate contaminant observed in the other animal mummies.

6.5 DISCUSSION

A summary of the results of this study is given in Table 6.2. The major products seen in the four animal mummies were either carbohydrate markers indicative of a sugar/gum, or degraded acyl lipids, which probably derive from animal fats or plant oils. Given that the degraded fats or oils are seen in or around the wrappings and not in direct contact with the body, they most likely derive from their intentional application as part of the embalming

process. The quantities involved and the sample locations chosen make contamination unlikely. Whereas the acyl lipids appeared to be of plant origin in most of the human mummies, the fatty acid distributions of the animal mummies were indicative of both animal (low abundance of $C_{16:0}$ compared with $C_{18:0}$) and plant origins (high abundance of $C_{16:0}$ compared with $C_{18:0}$; see Fig. 6.12 a-h). The ibis mummy contained only trace amounts of fatty acids, being largely sugar based, although the $C_{16:0}$ to $C_{18:0}$ ratios of 4.3 and 4.4 for the two samples analysed clearly indicate a plant origin for these trace components. These contrast with the three samples of wrappings taken from the mummified cat, and all very similar, have $C_{16:0}$ to $C_{18:0}$ ratios of 1.5, 1.3 and 1.5 (see Fig. 6.12 e-g). Together with the cholesterol derivatives and saturated TAGs (with a high abundance of the $C_{18:0}$ acyl group) identified in these samples, they confirm an animal origin for these embalming agents. Yet the fourth sample from the cat, a red material packed into the ears, has a quite different $C_{16:0}$ to $C_{18:0}$ ratio (3.7) and this value, together with an absence of cholesterol markers, would indicate a plant origin. All the samples from the two hawk mummies have remarkably similar $C_{16:0}$ to $C_{18:0}$ ratios (see Fig. 6.12 a-d), though their origin is unclear, the ratios are very similar to those of the cat, but with no sterol compounds present this cannot be confirmed. Beyond this, the identification of the precise animal or plant origin based on fatty acids alone is not possible, due to the degradation of major unsaturated components. The three samples from hawk 52.55.46 (6.4.1) all contained an unusually high abundance of 9,10-dihydroxyoctadecanoic acid, which was in fact one of the major components in all three samples. Interestingly the *threo* isomer was the predominant isomer with very little *erythro* present, suggesting that oxidation of the monounsaturated fatty acid present in the original fat/oil, i.e. oleic, has not been accompanied by the stereomutation often observed (Gunstone et al. 1986). This unusual abundance may reflect the fact that these embalming agents may have undergone heat treatment prior to application. The high abundance of the 9,10-dihydroxyoctadecanoic acid in these samples, together with the relatively low $C_{16:0}$ to $C_{18:0}$ ratios and a significant amount of the $C_{18:0}$ 1-monoacylglycerol with very little $C_{16:0}$ 1-monoacylglycerol, would seem to confirm an animal origin for these samples. Hawk 52.55.47 (6.4.2) also contained 9,10-dihydroxyoctadecanoic acid in relatively high abundance, but with similar abundances of both *threo* and *erythro* isomers, suggesting either an alternative source to hawk 52.55.46 or perhaps more likely, stereomutation during the oxidation of the oleic acid (Gunstone et al. 1986). Again, an animal origin for the lipids in this hawk mummy (6.4.1.2) is probable. Notably however, the oxidation products observed in the human mummies, i.e. dicarboxylic acids (C_7 - C_9) and monohydroxycarboxylic acids (C_{18} with the

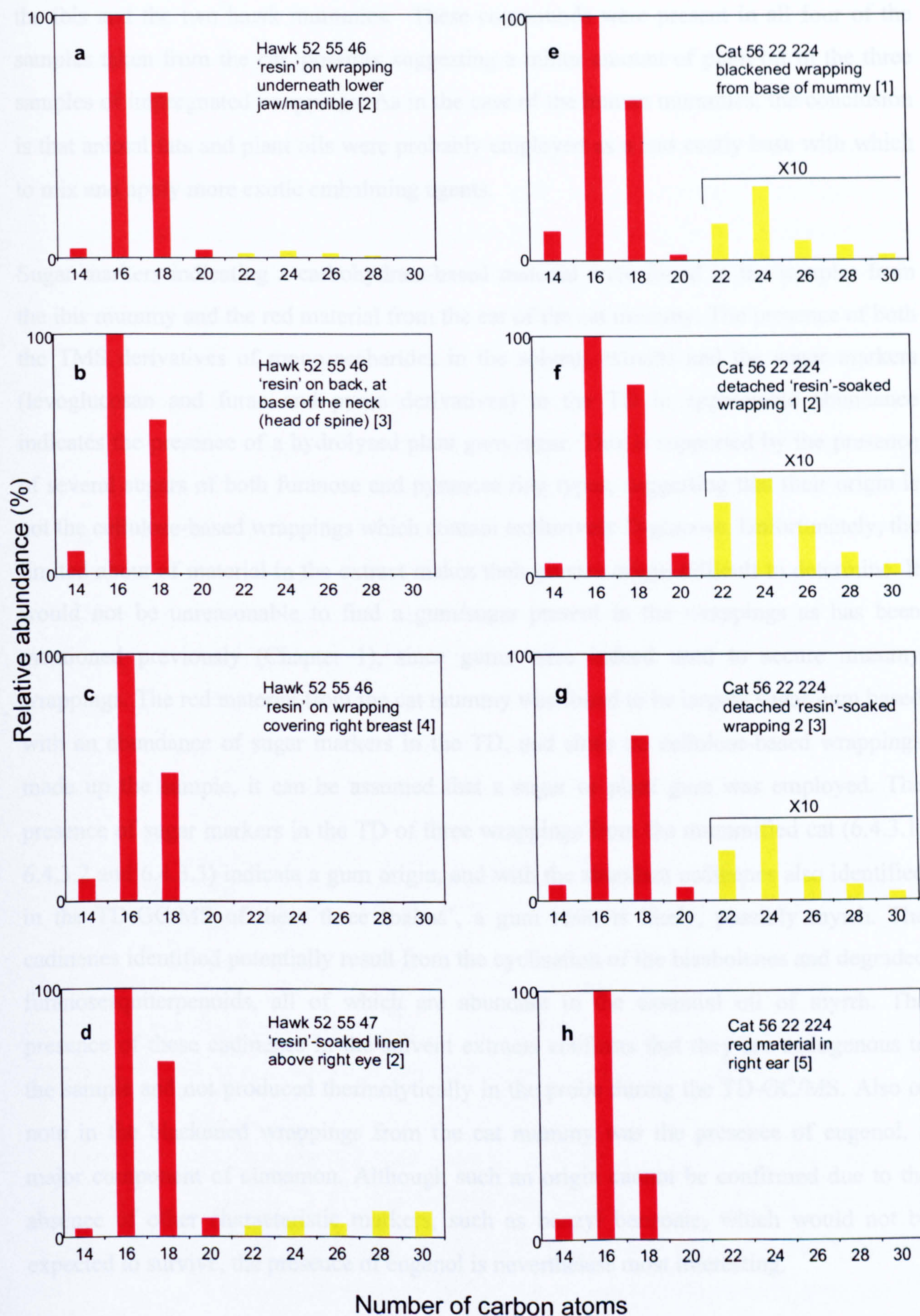


Figure 6.12 Histograms showing the distribution of fatty acids of even carbon number (fat/oil derived in red, beeswax derived in yellow) in the total lipid extracts and acid fractions from samples taken from the animal mummies.

hydroxyl group at C-8, C-9, C-10 and C-11, indicative of autoxidation) were absent from the ibis and the two hawk mummies. These compounds were present in all four of the samples taken from the cat, possibly suggesting a minor amount of plant oil in the three samples of impregnated wrappings. As in the case of the human mummies, the conclusion is that animal fats and plant oils were probably employed as a less costly base with which to mix and apply more exotic embalming agents.

Sugar markers indicating a carbohydrate-based material were found in the samples from the ibis mummy and the red material from the ear of the cat mummy. The presence of both the TMS derivatives of monosaccharides in the solvent extracts and the sugar markers (levoglucosan and furan and pyran derivatives) in the TD in appreciable abundance indicates the presence of a hydrolysed plant gum/sugar. This is supported by the presence of several sugars of both furanose and pyranose ring types, suggesting that their origin is not the cellulose-based wrappings which contain exclusively D-glucose. Unfortunately, the limited amount of material in the extract makes their precise origin difficult to determine. It would not be unreasonable to find a gum/sugar present in the wrappings as has been mentioned previously (Chapter 1), since gums were indeed used to secure mummy wrappings. The red material from the cat mummy was found to be largely sugar/gum based with an abundance of sugar markers in the TD, and since no cellulose-based wrappings made up the sample, it can be assumed that a sugar or plant gum was employed. The presence of sugar markers in the TD of three wrappings from the mummified cat (6.4.3.1, 6.4.3.2 and 6.4.3.3) indicate a gum origin, and with the abundant cadinenes also identified in the TD-GC/MS of these three 'balms', a gum resin is likely, possibly myrrh. The cadinenes identified potentially result from the cyclisation of the bisabolenes and degraded furanosesquiterpenoids, all of which are abundant in the essential oil of myrrh. The presence of these cadinenes in the solvent extracts confirms that they are endogenous to the sample and not produced thermolytically in the probe during the TD-GC/MS. Also of note in the blackened wrappings from the cat mummy was the presence of eugenol, a major component of cinnamon. Although such an origin cannot be confirmed due to the absence of other characteristic markers, such as benzyl benzoate, which would not be expected to survive, the presence of eugenol is nevertheless most interesting.

The diterpenoids characteristic of a coniferous resin were identified in the three impregnated wrappings from the mummified cat. As was the case in the human mummies, the diterpenoids are highly oxidised, 7-oxodehydroabietic acid being predominant with

lesser amounts of 15-hydroxy-7-oxodehydroabietic acid and relatively little dehydroabietic acid. Importantly, these diterpenoid were also accompanied by the sesquiterpenoids characteristic of cedar resin, identifying this product in an animal mummy for the first time (its identification based on their mass spectra, retention times and the presence of the naturally occurring tetramethyl-hexahydro-benzocycloheptadiene). As with the diterpenoid these sesquiterpenoids were highly oxidised, with 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadien-8-one and 3,5,5,9-tetramethyl-tetrahydro-2,4,9-benzocycloheptatrien-1-one dominant and the naturally occurring 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadiene a relatively minor component, demonstrating the importance of the characterisation and identification of oxidised biomarkers if an accurate picture is to be achieved. This latter natural compound characteristic of cedar has been identified in a number of Egyptian vessels, along with dehydroabietic and 7-oxodehydroabietic acids (Serpico & White 1998, p.1037-1048; Serpico 2000, p.463). This sesquiterpenoid would be susceptible to oxidation, and given the presence of the predominantly oxidised diterpenoids, i.e. 7-oxodehydroabietic acid, associated with these sesquiterpenoids then the oxidised forms of this substituted benzocycloheptadiene would indeed be expected to be the major components. The wrappings from the cat mummy contained significant quantities (2 to 5%) of coniferous terpenoids. Cedar produces relatively little resin compared to most other coniferous sources (Lucas 1989, p.319), and so is generally regarded as having been used relatively sparsely due to its lack of availability and expense.

“The cedar however, although it does produce resin when wounded, does not produce it readily or in great quantity, and, so for [sic] as is known to me, it has never been used as a source of resin, apart from its possible use in ancient Egypt now being considered, and, in my opinion, cedar may be excluded” (Lucas 1989, p.319).

The likely expense of imported cedar resin makes it clear that this cat mummy has been treated with extreme care and reverence, yet given that the ancient Egyptians had such high regard for animals, cats in particular, this should not be particularly surprising.

The use of beeswax, characterised chemically by *n*-alkanes (C₂₅ to C₃₃; Fig. 6.13 a-e), wax esters (C₄₀ to C₅₀; Fig. 6.13 f-j) and hydroxy wax esters (C₄₂ to C₅₄), was conclusively shown in the case of the cat mummy. In the three impregnated wrappings taken from the XXVIth to XXXth dynasty cat mummy (6.4.3) the solvent soluble extracts comprise 30-33% w/w beeswax (Figs. 6.3, 6.7 and 6.8), a notably narrow range for these three ‘balms’. The *n*-alkanes in the samples were typical of beeswax, with an odd-over-even preference and C₂₇ dominant. The wax esters were however less typical, with C₄₂ predominant although C₄₀ and C₄₆ were abundant. However the predominance of the C_{16:0} acyl group in

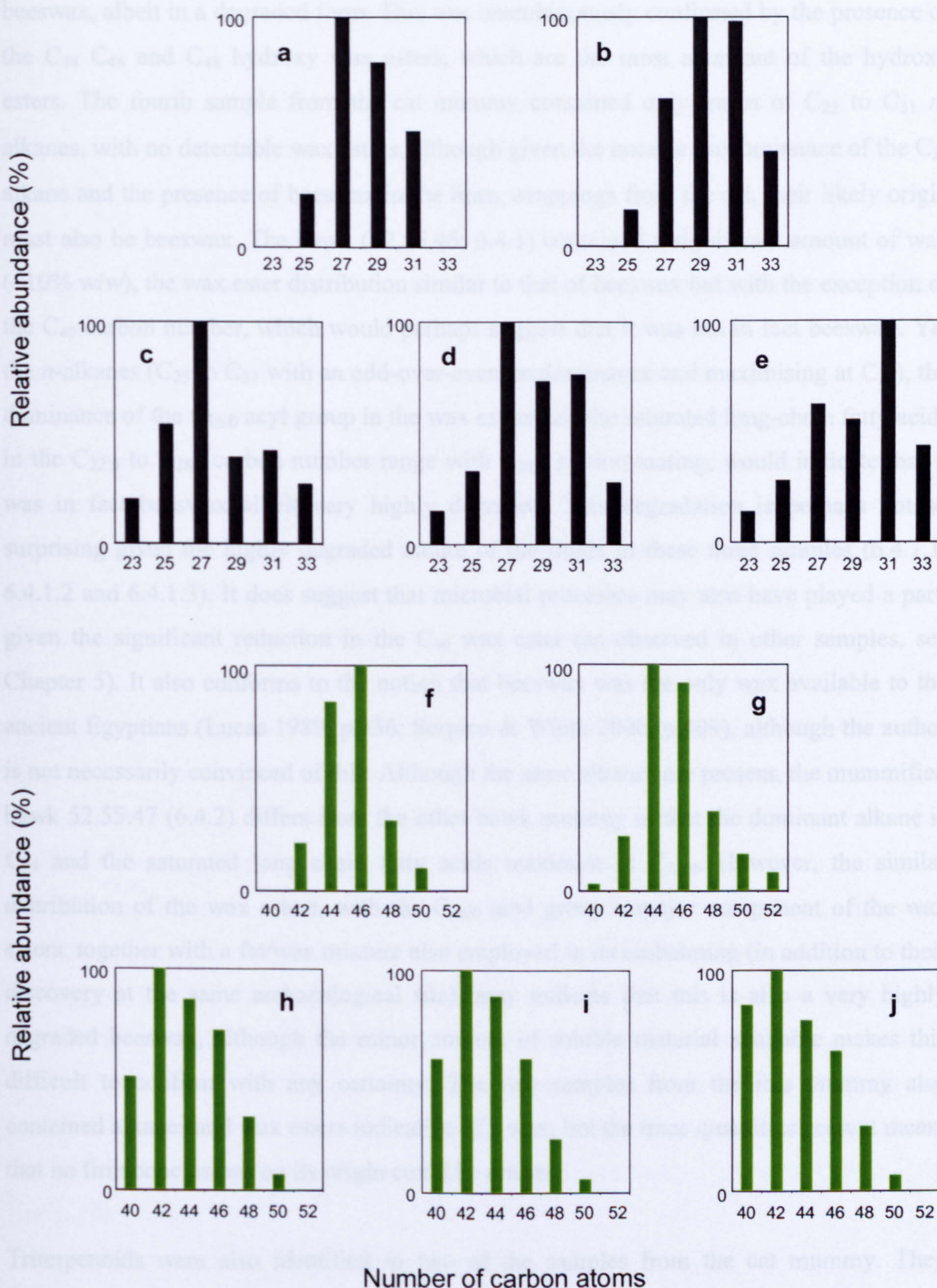


Figure 6.13 Histograms showing the distributions of: (i) hydrocarbons of odd carbon number in the total lipid extracts and neutral fractions from samples taken from the animal mummies containing beeswax (a to e); (ii) wax esters of even carbon number in the total lipid extracts and neutral fractions from samples taken from the animal mummies containing beeswax (f to j) [see Table 6.2].

these wax esters, together with the C₂₂ to C₃₀ free fatty acids, strongly suggest that this was beeswax, albeit in a degraded form. This was unambiguously confirmed by the presence of the C₄₄ C₄₆ and C₄₈ hydroxy wax esters, which are the most abundant of the hydroxy esters. The fourth sample from the cat mummy contained only traces of C₂₅ to C₃₁ *n*-alkanes, with no detectable wax esters, although given the notable predominance of the C₂₇ alkane and the presence of beeswax in the linen wrappings from the cat, their likely origin must also be beeswax. The hawk (52.55.46; 6.4.1) contained a significant amount of wax (~10% w/w), the wax ester distribution similar to that of beeswax but with the exception of the C₄₀ carbon number, which would perhaps suggest that it was not in fact beeswax. Yet the *n*-alkanes (C₂₅ to C₃₁ with an odd-over-even predominance and maximising at C₂₇), the dominance of the C_{16:0} acyl group in the wax esters and the saturated long-chain fatty acids in the C_{22:0} to C_{28:0} carbon number range with C_{24:0} predominating, would indicate that it was in fact beeswax albeit very highly degraded. This degradation is perhaps not so surprising given the highly degraded nature of the lipids in these three samples (6.4.1.1, 6.4.1.2 and 6.4.1.3). It does suggest that microbial processes may also have played a part, given the significant reduction in the C₄₀ wax ester (as observed in other samples, see Chapter 5). It also conforms to the notion that beeswax was the only wax available to the ancient Egyptians (Lucas 1989, p.336; Serpico & White 2000, p.409), although the author is not necessarily convinced of this. Although the same alkanes are present, the mummified hawk 52.55.47 (6.4.2) differs from the other hawk mummy in that the dominant alkane is C₂₉ and the saturated long chain fatty acids maximise at C_{32:0}. However, the similar distribution of the wax esters, with the C_{16:0} acyl group a major component of the wax esters, together with a fat/wax mixture also employed in its embalming (in addition to their discovery at the same archaeological site), may indicate that this is also a very highly degraded beeswax, although the minor amount of soluble material available makes this difficult to confirm with any certainty. The two samples from the ibis mummy also contained alkanes and wax esters indicative of a wax, but the trace quantities present meant that no firm conclusions on its origin could be drawn.

Triterpenoids were also identified in two of the samples from the cat mummy. They included moronic acid as the dominant component, with lesser amounts of dammaranes and a tentatively identified hydroxyoleanonic acid, along with nor- α -amyrene and nor- β -amyrene. The presence of moronic acid, which is diagnostic of the presence of *Pistacia* resin (Mills & White 1994, p.107-108), confirms the employment of this resin in the embalming of this cat. The sesquiterpenoid caryophyllene, not normally reported in aged

Pistacia, was identified (by both its mass spectrum and retention time in comparison with an authentic *Pistacia* 'standard') in the blackened wrapping, this being a major component of the resin from *Pistacia lentiscus* (Buckley & Evershed, unpublished data). Notably the blackened wrappings did not contain the euphane markers isomasticadienonic acid and masticadienonic acid identified in the Ptolemaic female mummy, yet despite the presence of relatively abundant dammaranes which might suggest a dammar origin, the presence of the relatively stable moronic acid clearly identifies *Pistacia* as the origin of the triterpenoids. Given the degraded nature of this sample, the resilience of moronic acid as the dominant component would tend to support the identification of dammar in the wrappings of the XXIInd dynasty mummy of Neskheon, since if *Pistacia* was present moronic acid would be expected to survive in these wrappings also [especially given the similar degree of oxidation in both samples (5.4.7.1; Fig. 5.10 and 6.4.3.1; Fig. 6.3)]. Norolean-17-en-3-one was not identified in the solvent extracts, indicating that the *Pistacia* resin was not subjected to excessive heating. The *Pistacia* content is significant in the blackened wrappings (6.4.3.1) and constitutes 4% of the extractable lipids, with only a minor quantity (0.2%) present in the 'resin'-soaked wrapping 2 (6.4.3.3).

The hydroxyaromatic acids observed in the majority of the human mummies were only found in the mummified cat. Their distribution is dominated by the 4-hydroxy-3-methoxybenzoic acid, with smaller amounts of p-hydroxybenzoic acid which is consistent with oxidation of unsaturated C-3 side chains (either acid or alcohol substituted) in the aromatic benzoate ester of balsamic resins or of ferulic acid present as a major component in *Umbelliferae* (Serpico 2000, p.450). The absence of the precursor compounds is not unexpected, given the susceptibility of these to the oxidation that is prevalent in mummies. A storax (*Liquidamber orientalis*) origin for these hydroxy aromatic acids can be ruled out, based on the absence of the cinnamic acids and triterpenoids dominant in storax (Pastorova et al 1998, p.1381-1393), the methoxy aromatic derivatives found being absent in storax but being major components of other balsamic resins (Pastorova et al 1998, p.1381-1393; Lucas 1989, p.95).

These results reveal the use of a mixture of commodities, with a compositional diversity greater than previously reported. They clearly demonstrate that commodities considered exotic were not restricted to human mummification as is often suggested (e.g. Lucas 1989, p.304; Spencer 1982, p.212). Despite the frequent use of the term 'bitumen' in connection with mummification, the notion that its general use in embalming has now been proven

“unequivocally” (Bahn 1992, p.109) can in no way be supported. In repeat searches the steranes and hopanes characteristic of petroleum bitumens (Rullkotter & Nissenbaum 1988, p.618-621) were not detected, leading to the conclusion that natural bitumens were not used in these animal mummies, contrary to the suggestion that this ‘inferior’ commodity (in comparison to resins, etc.) was more likely to have been used in animal mummification due to its relative cheapness (Lucas 1989, p.304). Furthermore, given that beeswax generally constitutes the greater proportion of the organic materials in the animal mummies as well as in humans, rather than the resins, the term embalming ‘wax’ also has at least equal validity to so-called ‘resin’ in both animal and human mummies. It is interesting to note that the Coptic (directly derived from the ancient Egyptian language) word for ‘wax’ is in fact ‘mum’ (Granville 1825). In the misguided belief that Egyptian mummification is now fully understood, these results clearly illustrate the dangers of making such assumptions about the materials the ancient Egyptians may have used to mummify both themselves and their animals.

6.6 CONCLUSIONS

The characterisation and identification of organic materials employed in the mummification of four animal mummies has been carried out using GC, GC/MS and sequential TD-GC/MS and Py-GC/MS. This investigation has resulted in a number of significant conclusions.

- (i) Sequential TD-GC/MS and Py-GC/MS have shown their value in aiding the identification of a wide range of organic materials including fats/oils, sugar/gum, cedar resin, *Pistacia* resin, and beeswax. For a fairly wide range of materials TD gave a useful ‘fingerprint’ which was comparable with the more conventional solvent extracts. However, polyfunctional components were not observed, and given the likely abundance of such polar molecules in aged samples this a notable limitation.
- (ii) The identification of a sugar/gum was demonstrated by the abundance of levoglucosan, furan and pyran derivatives in the TD-GC/MS.
- (iii) Bitumen was not present in these samples, as demonstrated by the absence of any bitumen markers in any of the samples analysed despite searches using mass chromatograms for the steranes (m/z 217) and hopanes (m/z 191) characteristic of natural bitumens.

- (iv) The identification for the first time of cedar resin in a mummy, most notably in an animal (cat) mummy from the Late Period (664-332 B.C.), as demonstrated by the presence of diterpenoids together with 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadien-8-one, 3,5,5,9-tetramethyl-tetrahydro-2,4,9-benzocycloheptatrien-1-one and 3,5,5,9-tetramethyl-hexahydro-3,9-benzocycloheptadiene characteristic of cedar.
- (v) The detection of *Pistacia* resin for the first time in an animal (cat) mummy from the Late Period (664-332 B.C.), as demonstrated by the presence of the moronic acid characteristic of *Pistacia*.
- (vi) The use of beeswax at least as extensively as resins, demonstrated by the presence of C₂₅ to C₃₃ alkanes, together with C₄₀ to C₅₀ wax esters and C₄₄ to C₄₈ hydroxy wax esters.
- (vii) The use of *Pistacia* and cedar resin mixed together with animal fat suggests that animal fat was not simply the cheap option to vegetable oil, but may in fact have had valued symbolic significance.
- (viii) Similar mixtures of commodities were found to those utilised in human mummies, corroborating the idea that animals were treated with similar reverence to humans rather than being mass-produced using inferior methods.

CHAPTER 7

Overview and future recommendations

CHAPTER 7: OVERVIEW AND FUTURE RECOMMENDATIONS

7.1: OVERVIEW

Chemical analyses have been used to characterise and identify the nature of the organic embalming agents employed in a total of eighteen mummies dating from 2600 BC to AD 400. The ability to characterise, identify and gain valuable chemical information from very small sample sizes (utilising sequential TD-GC/MS and Py-GC/MS) were key aspects of this research, an essentially non-destructive approach which is clearly attractive to museum curators, archaeologists and Egyptologists. Similarities and/or differences between the materials employed in human and animal mummies were also of great interest, as were the similarities and/or differences between materials used on various parts of the body. Both aspects are crucial in attempting to gain a meaningful understanding of the complex ritual nature and use of these organic materials. Using a ‘biomarker’ approach, the chemical characterisation and identification of these otherwise unidentifiable amorphous organic residues has provided significant insights into facets of an ancient culture over a 3,000 year period. A further aim of the research contained in this thesis was to enhance the understanding of the molecular transformations in these organic mummification materials, and to consider the use of conventional biomarkers in this context.

7.1.1 Historical and archaeological context

Although the nature of the organic embalming agents employed in ancient Egyptian mummification has long been disputed (as indeed have many aspects of the whole mummification process), there is nevertheless a widely held belief that there remains little to be discovered in a subject which some imagine is fully understood. Yet the suggestion that “*with the exception of a few minor details there is little about the ancient craft that is not well known*” (Mendelsohn 1944, p.1795) is quite simply nonsense.

Areas of particular contention include the use of either cedar or juniper and the use (or otherwise) of frankincense, myrrh and bitumen. The use of cedar in mummification has long been the subject of controversy (Baumann 1960, p.84-104; Lucas 1931, p.13-21; Mendelsohn 1944, p.1795-1804), the ‘oil of cedar’ referred to in secondary classical sources (see Chapter 1) now generally regarded as having been juniper oil. The actual

cedar resin was assumed to have rarely, if ever, been utilised due to the small amount of resin produced by *Cedrus libani*, making it nonviable in economic terms (Lucas 1989, p.319). Yet the finding of cedar in early dynastic jars (Serpico & White 1998, p.1041-1042) would clearly contradict this assumption. Although frankincense and myrrh are often mentioned in connection with mummification, the former has yet to be identified, and although the discovery of the latter has been reported (Coughlin 1977, p.7), it was not satisfactorily identified using the analytical techniques necessary to positively identify such a complex and easily degraded commodity. Again, bitumen was believed to have been little used (Lucas 1989, p.303), except in the mummification of animals as a 'cheaper' alternative to the 'resins' (Lucas 1989, p.304). However, in part as a result of more recent analysis (Connan & Dessort 1991, p.1445-1452; Rullkotter & Nissenbaum 1988, p.618-621; Connan 1999, p.33), the use of bitumen is currently regarded as having been widespread.

"The lid has, it seems, finally been put on a controversy about whether the ancient Egyptians used bitumen when mummifying the dead...J. Connan and D. Dessort show unequivocally that bitumen was indeed generally used in mummification, and far earlier than hitherto suspected" (Bahn 1992, p.109).

The relative treatment of humans and animals has also long been disputed, with the suggestion that animals are likely to have undergone less costly mummification (Lucas 1989, p.304). The many millions of animal mummies produced might indeed lead to the assumption that little care and expense was involved in their preparation in comparison with that of humans, and indeed it has been proposed that in the vast numbers of ibis and hawks in particular that they are likely to have been prepared simply "*by being immersed in vats of molten resin*" to both kill and preserve (Ikram & Dodson 1998, p.135-136), thereby implying that no real care was taken to mummify the animals concerned. Yet this cannot be assumed (see Chapter 1), since animals were generally treated with great respect and the embodiment of specific deities (see Table 1.1).

Since the ancient Egyptians left no written record of the embalming process, modern understanding must to a large extent be gleaned from the surviving mummies themselves. Yet the identity and precise use of the organic materials remains elusive, if only because of the few meaningful chemical studies carried out to date. Furthermore, the interest in these commodities is often seen as trivial and frivolous, despite their often crucial role in serving as both antibacterial agents (halting putrefaction of the bodies) and effective hydrophobic barriers, preventing moisture being reabsorbed [thereby obviating the reinitiation of bacterial (and chemical) decay]. This latter point is based on the misapprehension, still

widely held (David 1997, p.29), that dehydration [which is assumed (obviously misguided) to be irreversible] with natron is all that is necessary to effect a well preserved mummy which will stand the test of time, with the “oils” and “resin” “... only cosmetic treatments...” – clearly a nonsense. It is in the light of all these points that the research contained in this thesis must be considered.

7.1.2 Analytical approach

The techniques of sequential TD-GC/MS and Py-GC/MS and GC/MS following solvent extraction procedures were found to be complementary in the identification and determination of the characteristic compounds and their molecular transformations. Sequential TD-GC/MS and Py-GC/MS were shown to have significant potential, given their ability to analyse very small sample sizes (< 0.1 mg) and to determine both free and bound biomarkers. The nature of the organic commodities would suggest that both free and polymeric components are likely to be present (e.g. the free communic acids and polycommunic acid in juniper). This dual approach, conveniently analysing samples for both ‘free’ and ‘bound’ components, with minimal sample preparation required, and which facilitates the relatively rapid screening of a very large number of samples, is clearly attractive. Sequential TD-GC/MS and Py-GC/MS provided useful ‘fingerprints’, which could help in the identification of the material analysed. Notably beeswax, with its relatively involatile wax esters, was still able to be characterised and identified. Yet it should be noted that this methodology does have some notable shortcomings. Polyfunctional components of the samples, which are often prevalent in archaeological samples (especially in mummified remains), could not be observed using TD/Py-GC/MS alone. The derivatisation necessary to make these species amenable to GC was not carried out prior to TD/Py, since these highly oxygenated species can undergo undesirable side reactions with the methylating reagents normally used (eg. TMAH), resulting in several species for each original compound and potentially masking other components present. Therefore the use of solvent extraction procedures followed by the more conventional GC/MS can provide valuable complementary information about these important polyfunctional components which cannot be obtained by using TD in isolation. Given the complex nature of the material likely to have been used during the mummification process, this dual approach is desirable if the full elucidation of these commodities is to be achieved.

7.1.3 Molecular preservation and chemical transformations

The samples analysed were generally highly oxidised materials. The dominance of free fatty acids in most of the samples indicates the presence of hydrolysed acyl lipids, confirming an animal fat or plant oil. In many cases the biomarkers often used to characterise particular substances had themselves undergone significant chemical alteration, resulting in their absence or their presence as a trace component only. Two important examples of these are (1) dehydroabietic acid, which had been converted to its 7-oxo- and 15-hydroxy-7-oxo- derivatives, and (2) tetramethylbenzocycloheptadiene, converted to the 8-oxo compound. It is clear that in any attempt to identify the organic materials, to focus only on widely-known biomarkers would be insufficient, with major compounds in these embalming agents still to be elucidated. Certainly these materials have often undergone extensive chemical alteration. Particular materials, such as the mud packing in the mummy of Horemkenesi (Chapter 4), appear to facilitate extensive polymerisation, the packing containing very little extractable material but an appreciable abundance of 'bound' markers indicative of a highly polymerised acyl lipid. Extensive oxidation of the unsaturated fatty acids in many of these samples had also taken place, as evidenced by the presence of abundant dicarboxylic and mono- and dihydroxycarboxylic acids. The position of the vicinal hydroxyl group in the dihydroxy fatty acids gives the position of the original double bond in the unsaturated fatty acid, the presence of 11,12-dihydroxyoctadecanoic acid confirming the presence of 11-octadecenoic acid in the so-called 'resin' from the mummy of Horemkenesi (Chapter 4).

7.1.4 Determination of commodity use

As mentioned above, most samples were dominated by acyl lipids indicative of a fat or oil, the precise origins of which are difficult to determine. The more exotic commodities associated with embalming were observed in a number of the mummies investigated. Alkanes, wax esters and hydroxy wax esters confirmed the presence of significant quantities of beeswax in at least a third of the mummies. Diterpenoid acids were also identified and confirm the use of coniferous resins in eleven of the 18 mummies, although unlike beeswax it was used sparingly. The apparent use of a dammar resin in a XXIInd dynasty (945-715 BC) mummy was one of the most significant findings of this study, implying that Egypt had access to this south-East Asian commodity at a surprisingly early date. Future work, however, should include the radiocarbon dating of the triterpenoids of

this resin in order to eliminate the possibility of it resulting from 19th century conservation treatments. In addition, the finding, for the first time, of Chinese insect wax in a female mummy of Ptolemaic date confirms trade links with China (although quite possibly indirect) at that time – unsurprising given the post-Alexander the Great date.

Particularly notable was the first identification of cedar resin in a mummy, as demonstrated by the presence of diterpenoid acids and substituted benzocycloheptenes. This finding contradicts the assumption that the Egyptians are unlikely to have used it as a result of economic constraints, since ritual matters were clearly motivated by different, more ‘esoteric’ concerns. Furthermore, the finding of this presumably highly-prized and valuable resin in a mummified cat is of major importance, and clearly contradicts the notion that little care was afforded to animal mummies whilst supporting the notion that animals were treated with the same reverence as humans. The additional discovery of *Pistacia* resin in the same cat, as confirmed by the presence of triterpenoid acids, including moronic acid, also serves to re-emphasise the reverence in which cats in particular were held in ancient Egypt.

Second only to animal fat and plant oils in terms of the quantities employed, beeswax proved to be the major component of the ‘balm’ used on the mummies in this study, clearly seeming to contradict the general belief that “*the Egyptians only occasionally treated their dead with wax*” (Mendelsohn 1944, p.1795). Also of note was the absence of either true resins or gum resins in the samples taken from the mummy of Horemkenesi (Chapter 4) of the XXIst dynasty, a time when it is generally believed mummification achieved its highest standards with the extensive use of true resins. Yet degraded acyl lipids indicative of a plant oil or wax were the only components presents. Repeated searches failed to find the sterane and hopane biomarkers characteristic of bitumen, leading to the conclusion that natural bitumens were not used in any of the eighteen mummies studied. Given that the current study is by far the largest provenanced and dated study yet to be undertaken, the complete absence of bitumen only prolongs the uncertainty surrounding its use, and rather than finally ‘putting the lid on the controversy’ (see Bahn 1992, p.109), it has in fact been well and truly taken off again!

The terms ‘bitumen’ and ‘resin’ continued to be used widely and indiscriminately, with little regard for the wide variety of natural products which have clearly been used. Furthermore, given that beeswax rather than ‘resin’ generally constitutes the greater

proportion of the organic materials employed, the term embalming 'wax' has at least equal validity to the so-called 'resin' used in both animal and human mummies. It is interesting to note that the Coptic (directly derived from the ancient Egyptian language) word for 'wax' is in fact 'mum' (Granville 1825). In the misguided belief that Egyptian mummification is now fully understood, these results clearly illustrate the dangers of making such assumptions about the materials the ancient Egyptians may have used to mummify both themselves and their animals.

7.2 FUTURE RECOMMENDATIONS

7.2.1 Chemical characterisation of the organic embalming agents

The use of stable isotopes especially ^{13}C could potentially help in the identification of particular commodities, in particular the origin of the fatty acids present in these samples. Their origin could potentially be determined to differentiate between ruminant and non-ruminant or between C_3 and C_4 plants for example. Yet given the highly degraded nature of these materials (resulting in the absence of otherwise useful chemical indicators) together with the very wide range of oils and fats potentially employed (often as mixtures), it would only be through an extremely extensive study that meaningful results could be obtained, since a number of commodities may well have similar carbon values.

7.2.2 Radiocarbon dating

The use of ^{14}C dating, following preparative GC to minimise problems of contamination, could help in determining the origin of commodities, particularly where they are unusual or unexpected. The dammar identified in this research is a good example of the value of ^{14}C dating, since it would be of major importance for ancient trade routes if the resin was indeed ancient. Preparative GC, followed by ^{14}C dating, would differentiate between ancient plant products, such as resins and waxes, and conservation treatments carried out in the 19th and 20th centuries. A rigorous approach to problems of contamination would also be essential.

7.2.3 Sequential TD/GC/MS and Py-GC/MS

The continued use of sequential TD/GC/MS and Py-GC/MS is recommended for the characterisation and identification of samples from animal mummies in particular, given that sample availability for these is often difficult. Similarly the development of a derivatisation procedure which would not result in artefact compounds in order to facilitate the analysis of the polyfunctional compounds present would prove invaluable.

7.2.4 Determining nature and identity of the inorganic packing materials

In order to identify the nature of the inorganic packing materials, ICP/AAS/AES and ion chromatography might be used to help understand their role in the 'trapping' of volatiles and the formation of polymeric acyl lipids.

7.2.5 Use of reference materials

Reference materials might be used in order to aid identification of the constituents of the 'balms' given that the thermolytically altered and trimethylsilylated compounds are not commonly encountered.

7.2.6 Determining commodity use in a wider range of samples

The investigation of fully documented mummified remains with increased numbers of samples from individual mummies where feasible is strongly recommended, as is the study of a wider range in date, geographical origin and animal species. The analysis of mummies *in situ* would also limit the risk of contamination from sampling procedures and conservation treatments. All these considerations would allow a more extensive and meaningful picture to be obtained, ultimately providing ever deeper insights into important facets of ancient Egyptian culture.

CHAPTER 8

Experimental

CHAPTER 8: EXPERIMENTAL

8.1 GENERAL

8.1.1. Glassware

All reusable glassware was cleaned thoroughly by soaking in a solution of the detergent Micro[®], followed by rigorous manual cleaning. It was then rinsed thoroughly with double distilled water before oven drying. Prior to use, all glassware was rinsed with the appropriate solvent and oven dried before use.

8.1.2. Solvents

All solvents used were of either double distilled or HPLC grade (Fisons plc/Rathburn Chemical Company). Double distilled water was distilled on site.

8.1.3 Storage

All samples were stored in vials in darkness in an attempt to limit chemical degradation. Given the museum conditions in which the mummies were kept, i.e. room temperature, no steps were taken to refrigerate/freezer the samples. After solvent extraction, the total lipid extracts were kept in vials and placed in a desiccator in the dark. Derivatized samples of the total lipid extracts (TLEs), acid and neutral fractions and fractionated neutrals were discarded immediately after analysis.

8.1.4 Archaeological samples

All archaeological samples were kindly provided by Sue Giles of Bristol Museum, Dr. Jeffrey Spencer and Dr. John Taylor of the British Museum, Joanna Hayward of the National Museums and Galleries on Merseyside, Liverpool, Dr. Rosalie David of Manchester University Museum and Dr. Liz Goring, Dr. Kathy Eremin and Dr. Jim Tate of the National Museums of Scotland, Edinburgh.

8.1.5 Sample preparation

Samples for analysis were weighed and then ground under liquid nitrogen in a mortar and pestle until a fine powder. These ground samples were used for both solvent extraction procedures and sequential TD-GC/MS and Py-GC/MS in order that direct comparisons could be made between the two approaches. A flow diagram of the experimental protocol is shown in Figure 8.1.

8.2 EXTRACTION

With each sample set, analytical 'blanks' were also processed in order to monitor for any contamination from the analytical procedure (glassware, syringes, etc.).

8.2.1 Solvent extraction

Samples for analysis were weighed and then ground under liquid nitrogen in a mortar and pestle until a fine powder. A weighed amount of these ground samples (typically 50 mg depending on sample available) was taken, and where appropriate an internal standard was added for quantification (10-100 µg of *n*-tetratriacontane, *n*-C₃₄ alkane and 10-100 µg of *n*-heneicosanoic acid, *n*-C₂₁ fatty acid). The lipids were extracted with an appropriate volume (eg. 50 mg, 1ml) of chloroform methanol solution (2:1 v/v; 3 x 60 minutes sonication). After centrifugation (20 min, 2000 rpm) the supernatant solvent was removed from the residue and placed in a vial. The three extracts were combined and the solvent reduced by rotary evaporation. Following transfer of the TLEs to a screw-capped vial, the remaining solvent was removed by evaporation under a gentle stream of nitrogen at 40°C. The residue was dried over silica and re-weighed where appropriate.

8.3 SEPARATION

8.3.1 Acid/neutral separation

Aliquots of the TLEs (see 7.2.1) were separated into 'acid' and 'neutral' fractions using bonded aminopropyl solid-phase extraction cartridges (100 mg, 1 ml or 500 mg, 2.8 ml) (Varian). A cartridge was first pre-eluted with dichloromethane (DCM)/isopropanol (IPA) (5ml or 20ml) and activated with hexane (5 or 12 ml). Extracts, dissolved/dispersed in

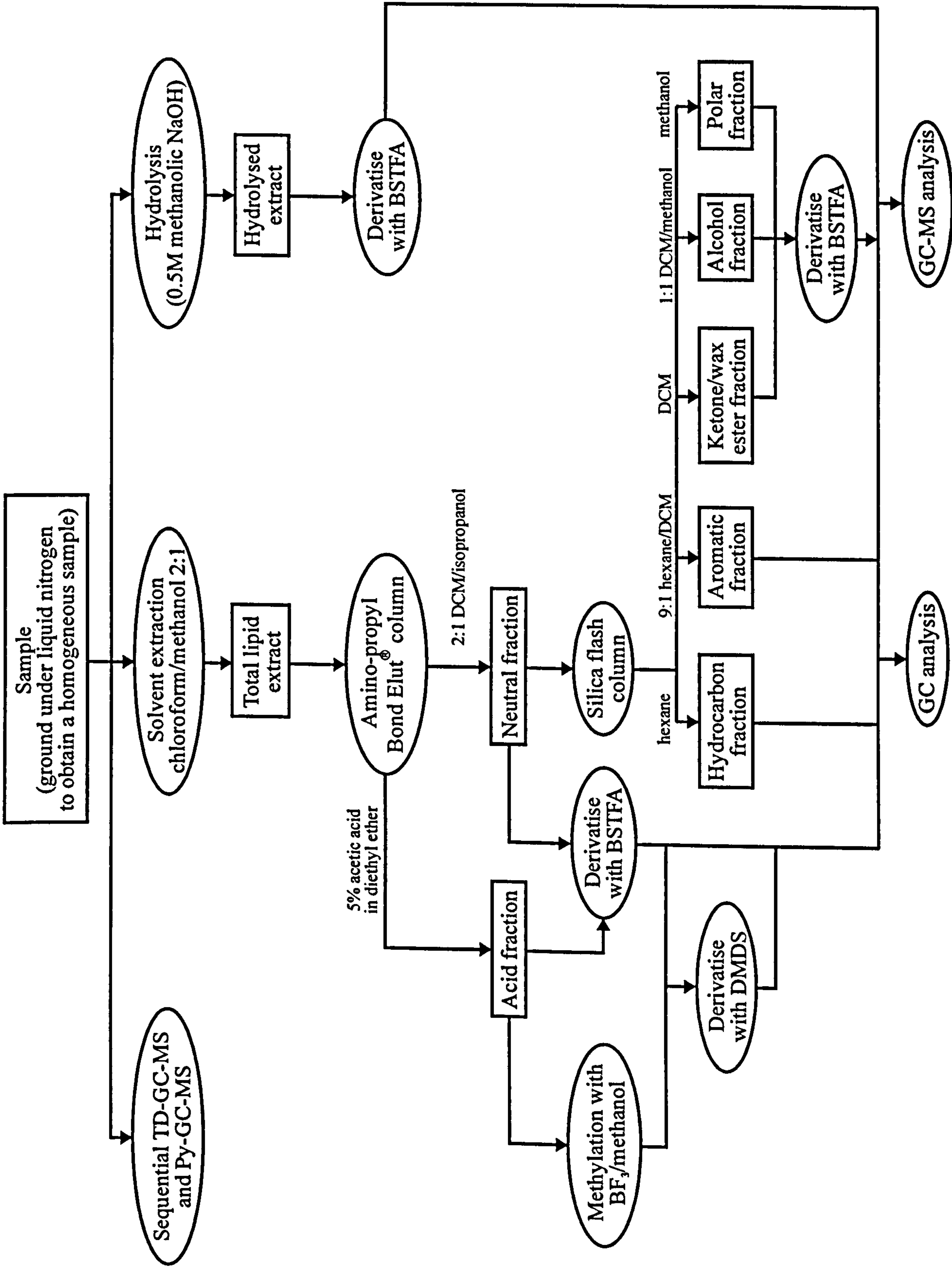


Figure 8.1 A schematic of the adopted experimental protocol.

DCM/isopropanol (2:1 w/w), were flushed through the cartridge. The 'neutral' fraction was collected by elution with 2:1 v/v DCM/IPA (3 or 12 ml), and flushing the cartridge with 5% acetic acid in diethylether (3 or 12 ml) resulted in the elution of the 'acid' fraction. Both fractions were evaporated under reduced pressure, transferred to vials and the solvent removed under nitrogen at 40°C.

8.3.2 Fractionation of neutral lipids

Column chromatography was used to separate the neutral fractions further (where appropriate). Columns were packed with a slurry of activated silica gel 60 (160°C, >24 hour; Fluka) (0.4 g) in hexane. Each column was pre-eluted with hexane. Neutral fractions (5-10 mg) were dissolved/dispersed in hexane and added to the top of the column. Gradient elution was performed under positive pressure supplied by a stream of nitrogen. The eluents used comprised five separate solvent systems of increasing polarity, ie. hexane, hexane/DCM (9:1), DCM, DCM/methanol (1:1 w/w) and methanol. These gave the hydrocarbon fraction, aromatic fraction, ketone/wax ester fraction, esters/alcohol fraction and polar fraction respectively. The elution volumes used were 3.0, 1.5, 2.0, 3.0, 2.5 ml for solvents in order of increasing polarity. Column fractions were collected in small vials and the solvent removed under a gentle stream of nitrogen at 40°C.

8.4 CHEMICAL TREATMENTS AND DERIVATISATION

8.4.1 Hydrolysis

Samples (5-50 mg) were added to Teflon-lined screw-capped test-tubes and mixed with a methanolic solution of 0.5M sodium hydroxide (1-2 ml depending on sample size). These were heated in a water bath at 70°C for 2 h. After cooling, the solution was acidified with 4M hydrochloric acid to pH 3 (pH paper) and 1-2 ml of water (double distilled) added. Samples were then extracted with diethyl ether (3 x 3-5 ml) (DCM was also tried but was insufficiently polar for the extraction of the polyfunctional compounds present in the archaeological samples). The combined ether extracts were washed with water until neutral (2 x 3-5 ml). The combined ether extracts were then passed through an anhydrous sodium sulphate column to remove the majority of the residual water and that remaining removed by drying over magnesium sulphate ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$). These ether extracts were filtered through a plug of cotton wool (solvent extracted) to remove any particulate matter

(MgSO₄.H₂O). Rotary evaporation under reduced pressure was used to remove the majority of the ether before transferring the extracts to vials and evaporating the remaining ether under nitrogen at 40°C.

8.4.2 Trimethylsilylation

Free hydroxyl and carboxylic acid groups from total lipid extracts, 'acid' fractions, 'neutral' fractions and neutral lipid fractionations were derivatised to their corresponding trimethylsilyl (TMS) ethers and esters, respectively, using *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA, Sigma) containing 1% trimethylchlorosilane (30-50 µl, 70°C, 1 h). BSTFA was then removed under a gentle stream of nitrogen and the derivatised sample redissolved in hexane or dichloromethane and analysed immediately by GC or GC/MS.

8.4.3 Methylation

Fatty acids were methylated by addition of 1 ml 14% w/v boron trifluoride methanol complex (70°C, 1 h, Aldrich). After cooling, 5 ml double distilled water was added to quench the reaction and the fatty acid methyl esters (FAMES) extracted into DCM (2 x 6 ml). DCM was removed under a stream of nitrogen and the methyl esters redissolved in DCM or hexane.

8.4.4 Dimethyl disulphide derivatisation of unsaturated fatty acids

DMDS (100 µl, Aldrich) and iodine in diethyl ether (6% w/v, 20 µl) were added to a solution of the fatty acid methyl esters in hexane (100 µl). This mixture was heated at 50°C for 24 h (in the dark). Samples were diluted with DCM (200 µl), water (200 µl) added and the excess iodine reduced by treatment with aqueous sodium thiosulphate (5% w/v, 200 µl). The organic phase was removed with a syringe and the aqueous phase extracted a second time with DCM (200 µl). The combined DCM extracts were diluted (with DCM) to the appropriate concentration for GC and GC/MS analysis. Analysis was carried out immediately to avoid the problem of decomposition of the DMDS adducts.

8.5 INSTRUMENTAL ANALYSIS

8.5.1 Elemental analysis

Elemental analyses were performed using a Perkin Elmer 240C elemental analyser to determine total carbon, hydrogen and nitrogen composition of analytes. Inorganic carbon content was determined using a Strohlein Instruments Coulomat 702 carbon analyser adapted to analyse CO₂ liberated from H₃PO₄ digestion; the TOC value was then calculated as the difference between total carbon and total inorganic carbon. Each sample was analysed a total of four times and a mean TOC value calculated; values generally exhibited errors $\leq \pm 0.1\%$ _{dwt}.

8.5.2 Thermal desorption-gas chromatography-mass spectrometry (TD-GC/MS) and pyrolysis-gas chromatography-mass spectrometry (Py-GC/MS)

A CDS 1000 Pyroprobe (Chemical Data System, Oxford, PA, USA) unit fitted with a platinum coil probe was used for the thermal extraction of free biomarker compounds and the pyrolysis of bound/polymerised components from the ground samples (50-250 µg of sample weighed using a microanalytical balance). The samples were loaded into quartz tubes plugged with solvent extracted glass wool. The quartz tubes were then inserted into the platinum coil of the probe which was inserted into the heated injector of a GC interfaced to the mass spectrometer. The quartz tubes were pre-cleaned by heating them in a furnace at 600°C for 24 h. They were also heated to 1000°C in the platinum coil probe prior to use. The TD temperature was 310°C (held for 10 s) and the Py temperature 610°C (held for 10 seconds). TD-GC/MS and Py-GC/MS were carried out on a Carlo Erba (Milan, Italy) 4130 GC fitted with a fused silica capillary column (50 m x 0.32 mm id) coated with a dimethyl polysiloxane bonded stationary phase (CP Sil-5 CB, 0.4 µm film thickness; oven temperature programme, 35°C (5 min) to 320°C (15 min) at a rate of 4°C min⁻¹) interfaced to a Finnigan (Sunnyvale, CA, USA) 4500 mass spectrometer operated in full scan mode (40-650 Da, 1 scan s⁻¹; electron energy, 70 eV; filament current, 300 µA; source temperature, 170°C). The Pyroprobe interface temperature was 280°C and the transfer line temperature from the GC to the mass spectrometer was 290°C. Helium was used as carrier gas. Peaks were identified based on both their mass spectra (NIST/EPA/NAH Mass Spectral Database) and retention times.

8.5.3 Gas chromatography (GC)

All total lipid extracts, 'acid' and 'neutral' fractions were first screened on a Hewlett-Packard 5890 Series II GC equipped with a fused-silica capillary column (15m x 0.32 mm) coated with DB-1 (film thickness 0.1 μm). Derivatised total lipid extracts, acid fractions and neutral fractions (1.0 μl) dissolved in DCM were injected on-column. The temperature was held isothermally for 2 min at 50°C then programmed from 50 to 350°C, and a rate of 10° min⁻¹ and held at 350°C for 10 min. The flame ionisation detector (FID) was set at a temperature at 350°C. Hydrogen was used as a carrier gas and maintained at a head pressure of 10 psi.

The 'acid' fractions (where separation of components was poor) and hydrolysed extracts were analysed on a Hewlett-Packard 5890 Series II GC equipped with a fused-silica capillary column (50 m x 0.32 mm) coated with a 100% polymethyl siloxane stationary phase (CPSil-5 CB, film thickness 0.12 μm). Derivatised fractions (1.0 μl) in dichloromethane were injected on-column. The temperature was programmed from 50°C (2 min) to 200°C at 10 min⁻¹, then 200 to 300°C (10 min) at 4° min⁻¹. The FID was set at a temperature of 300°C. Hydrogen was used as a carrier gas and maintained at a head pressure of 10 psi.

FAMES were analysed on a Hewlett-Packard 5890 Series II GC equipped with a fused-silica capillary column (25 or 50m x 0.32 mm i.d.) coated with 70% cyanopropyl equivalent modified siloxane (BPX70 'non-polar', 0.12 μm film thickness). Prepared samples dissolved in hexane or DCM (1 μl) were injected on-column. The temperature programme ran from 50°C (1 min) to 150°C at 15°C min⁻¹, then from 150 to 230°C at 4°C min⁻¹ and held at this temperature for 20 min. Helium was used as the carrier gas at a head pressure of 10 psi. DMDS derivatives of the unsaturated fatty acid methyl esters were analysed using the same conditions and set up employed for the analysis of the FAMES.

8.5.4 Gas Chromatography/Mass Spectrometry (GC/MS)

GC/MS analyses were performed using a Carlo Erba 5160 Mega Series GC, equipped with on-column injection, connected to a Finnigan 4500 quadrupole MS *via* a heated transfer line. The MS was set to scan in the range m/z 50-850 in a cycle time of 1.5 s (triacylglycerols and wax esters) or m/z 35-650 in a cycle time of 1.0 s (all other samples)

and was operated with an electron ionisation potential of 70 eV. The capillary column and temperature programmes employed were identical to those described for respective GC analyses (see 7.5.3). Helium was used as the carrier gas, the electron energy maintained at 300 μ A and the ion source temperature was 170°C. Peaks were identified based on both their mass spectra (NIST/EPA/NIH Mass Spectral Database) and retention times.

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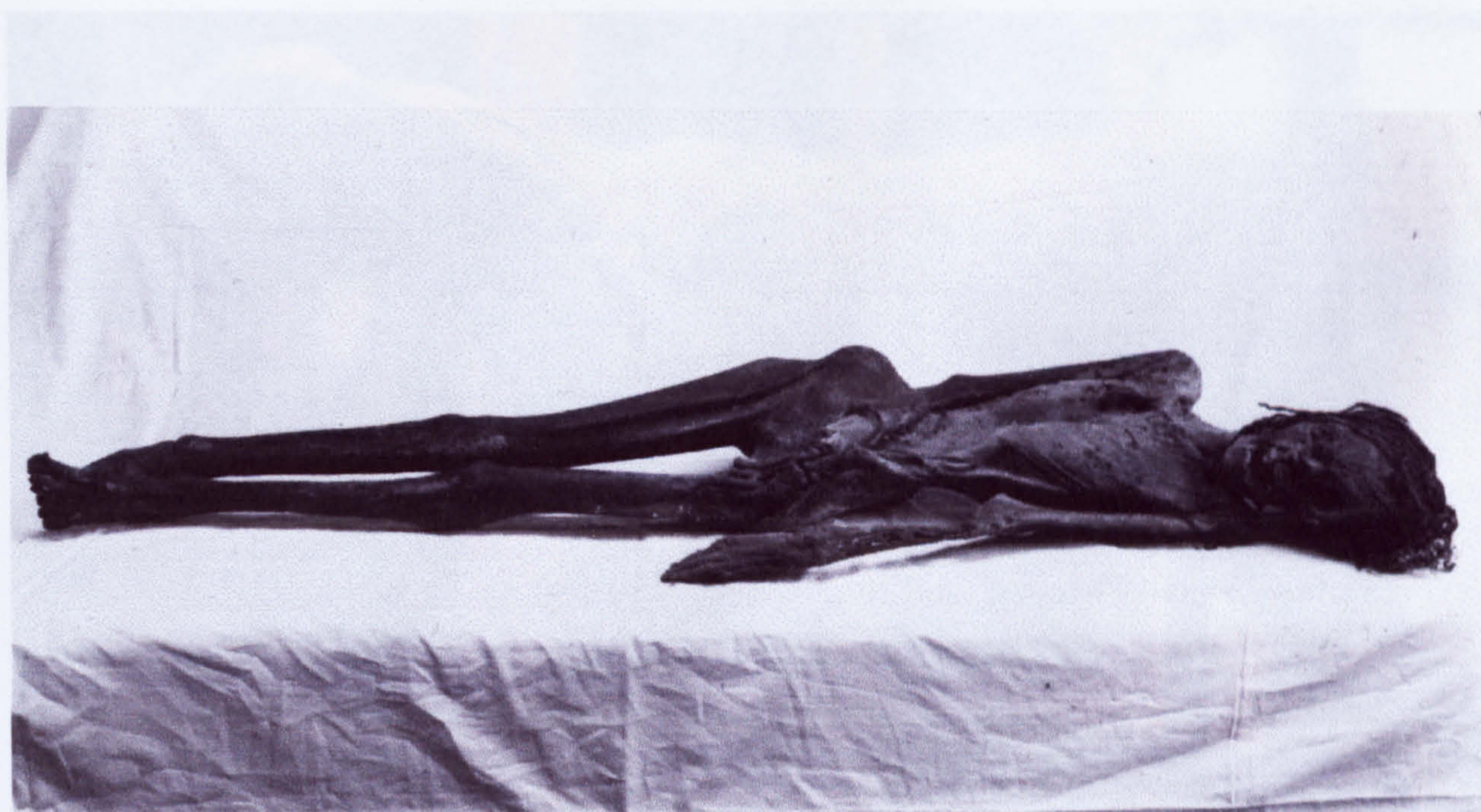
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III.1 Burial of female adult, Hierakonpolis Cemetery HK.43, c.3400 BC, Predynastic Period (S.A. Buckley)



III. 2 Burial of adult male showing early attempts at mummification, Sakkara, 2890-2686 BC, IInd dynasty (after Smith & Dawson 1924, fig.1)



III.3 Mummy of Queen Ashayet shortly after discovery, Deir el-Bahari, c.2000 BC, XIth dynasty (courtesy of Metropolitan Museum of Art New York)

III.4 Mummy of domesticated dog of Amenhotep III?, Valley of the Kings (KV 50), c.1400 BC (S.A. Buckley)



III. 4 Mummy of domesticated dog of Amenhotep II(?), Valley of the Kings (KV.50), c.1400 BC (S.A. Buckley)



III.5 Mummies of Yuya and Tuya, parents-in-law of Amenhotep III, Valley of the Kings (KV.46), c.1360 BC (after Quibell 1908, pl.LVII-LVIII, LIX-LX)



III. 6 Mummy of Seti I, Deir el-Bahari (DB.320), 1279 BC, XIXth dynasty (after Harris & Weeks 1973, p.18)



III. 7 Mummy of Horemkenesi, Deir el-Bahari, c.1040 BC, XXIst dynasty (courtesy of Bristol Museum)



III. 8 Mummy of Nesitanebasheru, Deir el-Bahari (DB.320), XXIst-XXIInd dynasty (after Smith 1912, pl.LXXXVIII)



III. 9 Mummy of Pasjenyentaihet, unprovenanced, C.1st BC-AD C. 1st, Leiden Rijksmuseum (S.A. Buckley)



Ill. 10 Wrapped and unwrapped mummies of two male children, Thebes, c.30 BC-AD 395, Roman Period (courtesy of Trustees of the National Museums of Scotland)